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THE NUCLEAR THERMAL EFFECTS ON HUMIDITY SATURATED COMPOSITE **MATERIALS**

Rockwell International Corporation Los Angeles Division P.O. Box 92098 Los Angeles, California 90009

February 1979

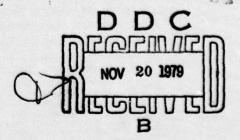
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SUMMARY

The primary objective of The Nuclear Thermal Effects on Humidity Saturated Composite Materials program was the development of composite design data in the crucial area of nuclear hardness after humidity saturation.

This objective was accomplished by testing over 4,000 specimens. These test specimens were constructed from 14 different common aerospace structural composite materials, including fiberglass reinforced epoxy and polyimide systems, quartz reinforced epoxy and polyimide systems, graphite-reinforced epoxy and polyimide systems, Kevlar reinforced epoxy systems, and a boron reinforced epoxy system. Each material was tested at four common structural thicknesses. The nuclear thermal energy levels were 15, 20, 25, and 30 calories per square centimeter (cal/cm²) absorbed. Specimens were tested in a dry condition and after humidity saturation at 160° F, which simulated a hot humid exposure in a field condition such as Guam. This program demonstrated that the nuclear thermal hardness of organic matrix composites is significantly degraded by humidity saturation. This test program also indicated that there can be significant differences in the nuclear thermal hardness of resin systems which are qualified to the same military specification. This test program demonstrated that reinforcement has a significant effect on the damage threshold level for any given resin system. It was shown that at the saturation level, substantial degradation of the surface coating will occur. This degradation manifests itself by chipping, blistering, and discoloration of the reflective coating after nuclear thermal radiation exposure. The damage level is proportional to the moisture content at saturation.

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PREFACE

This report describes the investigation conducted to determine the effects of humidity saturation on the nuclear thermal hardness of 14 common aerospace structural composite materials.

This work was funded by the Defense Nuclear Agency under contract DNA001-78-C-0062. The contract officer's representitive was Captain Mike Rafferty. The period of performance was from December 1977 to February 1979. This work was sponsored by the Defense Nuclear Agency under RDT&E RMSS code B3420 78464 N990QA X AJ 505-01 H2590D.

The author wishes to thank Captain Mike Rafferty for his guidance and support during the performance of this contract. The author would also like to acknowledge the Air Force Materials Laboratory (AFML) and the University of Dayton, for its fine support while operating the DNA Tri-Service Thermal Radiation Test facility. The author wishes to extend a special thanks to Mr. Nick Olson, of the University of Dayton, for his diligent efforts in testing over 4,000 specimens for this program.

This was a technology study of nuclear thermal flash damage on humidity-saturated composite materials. The generic composites used are typical of composite structures on all current and future aircraft, both commercial and military.

BACKGROUND

Due to advances in nuclear weapons technology, the use of tactical nuclear weapons as a defensive threat against military aircraft is becoming increasingly probable. This, coupled with the existing strategic offensive first-strike threat, deems it increasingly important for these aircraft to be hardened against the environments produced by an endoatmospheric nuclear explosion. These hardening factors should be considered at all stages of aircraft systems design and development.

The state-of-the-art development of nonmetallic materials suitable for primary/secondary structures of future aircraft is advancing rapidly. The use of these materials has been demonstrated on aircraft and missiles. The cost/weight advantages accrued through their use strongly indicates a much broader application in future weapon systems. The design of a successful structural aircraft component requires that the designer has access to all applicable material properties. To build a sound design, he must use all building blocks of proper design, mechanical and physical properties, and environmental effects on these mechanical and physical properties.

Fiber-reinforced organic matrix composites are common design elements for lightweight structures on modern aircraft. These organic matrix composites can be used as skins or face sheets in honeycomb structures or as laminates in monocoque structures. In spite of increasing use of organic matrix composites on modern air vehicle structures, little design data exist on their performance in the thermal environment produced by the endoatmospheric detonation of a nuclear weapon.

Fiber- or filament-reinforced organic matrix composite structures absorb from 1- to 3- percent water, depending on the resin system and reinforcement used. This humidity saturation has been shown to severely degrade some mechanical properties, as indicated in MIL-HDBK-17. Until recently, all nuclear thermal pulse testing was accomplished on unsaturated specimens. This data base has been used in all nuclear survivability designs and analyses on composite parts. Recently, Rockwell ran thermal pulse exposure tests on several humidity-saturated composite parts. All test parts were fabricated per applicable specifications and cut to test specimen configurations. Six test specimens were cut from each part. Three specimens were used as controls, and three were placed in an MIL-STD-810 humidity chamber for saturation at 160° F. All specimens were coated with white aliphatic polyurethane per MIL-C-83286 which overcoated MIL-P-23777 zinc chromate primer. All specimens were exposed to a B-1-type pulse shape and 20 cal/cm² absorbed. All unsaturated control specimens survived multiple exposures without degradation. All humidity-saturated specimens suffered severe degradation after one thermal pulse. After multiple thermal pulse exposures, the saturated graphite/epoxy composite

specimens were catastrophically degraded. This degradation could severely limit the use of composite parts in nuclearly hardened systems.

During this program, Rockwell demonstrated this nuclear hardness reduction in a broad range of fiber-reinforced organic matrix composite systems.

Rockwell's Los Angeles Division (LAD) used its unique experience to conduct this 14-month program in support of the Defense Nuclear Agency's investigation of the nuclear thermal effects on humidity-saturated composite materials when subjected to a variety of nuclear thermal pulses, simulating those produced from the endoatmospheric detonation of nuclear weapons.

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INTRODUCTION

This program was conducted in an effort to produce experimental data demonstrating the response of humidity-saturated, fiber-reinforced, organic matrix composites in the thermal flash environment produced by the endoatmospheric detonation of a nuclear weapon.

The resin materials used for evaluation as part of this program were epoxy and polyimide. Fiberglass, quartz, graphite, Kevlar, and boron were evaluated as typical reinforcements. The composite systems evaluated were those in common use on military hardware. All specimens were fabricated in accordance with the appropriate specification. All prepreg materials used were procured from the same suppliers and in accordance with appropriate specifications for the composite material used.

Over 4,000 specimens were fabricated and tested during this program. All specimen fabrication was performed at LAD. All specimen testing was performed at the DNA Tri-Service Thermal Radiation Test Facility at Wright-Patterson Air Force Base, Dayton, Ohio. Six specimens were tested at each test condition; three were tested in a dry condition, and three were tested after humidity saturation at 160° F and 90-percent relative humidity.

Section II

SPECIMEN FABRICATION

MATERIALS

Rockwell conducted a test program to determine the effects of humidity saturation on nuclear thermal hardness of fiber-reinforced, organic matrix composite systems. LAD determined the effects of face sheet or laminate thickness on degradation levels, and the damage threshold for each system in relation to absorbed energy and multiple exposures. This assessment was performed on both humidity saturated and dry specimens of each system. The organic resin-matrix systems used were epoxy and polyimide. The fiber reinforcements tested were fiberglass, quartz, graphite, Kevlar, and boron. The composite systems selected were those in current use on military and commercial air vehicles.

Six test specimens for each test condition and each composite configuration in common industry use were fabricated.

EPOXY SYSTEMS

Epoxy systems are as follows:

- 1. 7781 fiberglass preimpregnated with F-161 resin from Hexcel Corporation and CE-9000 resin from Ferro Corporation
- 2. 581 quartz preimpregnated with F-161 and resin from Hexcel Corporation
- 3. T300 graphite fiber from Union Carbide Corporation preimpregnated with 5208 resin from the Narmco Division of Celanese Corporation, type AS graphite fiber from Hercules Corporation preimpregnated with 3501-5A resin from the Hercules Corporation, and 934 resin from the Fiberite Corporation
- 4. Boron filament tape from the 3M Corporation

ADDITIONAL POLYIMIDE SYSTEMS

Additional polyimide systems are as follows:

1. 7781 fiberglass preimpregnated with F-178 resin from Hexcel Corporation and 2272 resin from Ferro Corporation

- 2. 581 quartz preimpregnated with F-178 resin from Hexcel Corporation
- 3. Type AS graphite fiber from Hercules Corporation impregnated with F-178 resins from the Hexcel Corporation

The pulse shape simulated a strategic weapon. The peak flux was 55 cal/cm^2 per second. That flux peak was held constant throughout the program. The fluence was varied from 15 to 30 cal/cm² by varying the time at the peak of the pulse.

Each fiber-reinforced, organic resin-matrix composite was tested at four energy levels; i.e., 15, 20, 25, and 30 cal/cm². Six specimens at each energy level were tested with one, three, and five pulses.

To determine the effects of heat sink, thickness, and temperature rise, each material was tested at four common structural thicknesses; i.e., three, four, five, and six plies.

Three specimens of the six tested at each condition were humidity saturated at 160° F and 90-percent relative humidity.

Six test specimens were required for each test condition. Three energy levels multiplied by four different pulse requirements equals 12 test conditions multiplied by six specimens per condition equals 72 specimens times four structural thicknesses for a total of 288 specimens per material, multiplied by 14 different materials, for a total of 4,032 specimens fabricated and tested under this program.

To simulate air vehicle structure, all precured laminates were bonded to 1/4- by 3/16-inch cell, 4-pound density, HRP honeycomb core. The laminates were bonded with AF-143 high-temperature epoxy adhesive manufactured by the 3M Corporation. All test specimens were primed with MIL-P-23777 zinc chromate primer. They were finished with highly reflective ensignia white aliphatic polyurethane per MIL-C-83286. Each face sheet or laminate was fabricated using the appropriate military or company specification to produce high-quality structural laminates.

Section III

EXPERIMENTAL TEST DATA

SYSTEM 1

System 1 was a 7781 style fiberglass-reinforced, epoxy-novalac, resinmatrix composite manufactured by Hexcel Corporation. This material is sold under the Hexcel trade name of F-161 epoxy with 7781 fiberglass reinforcement.

15 CAL/CM² ABSORBED^a

System 1 was unaffected by the 15 cal/cm^2 energy level at any structural thickness and up to five flashes. The humidity saturation had no effect at this energy level.

20 CAL/CM²

System 1 was unaffected by the $20~\text{cal/cm}^2$ energy level at any structural thickness and up to five flashes. The humidity saturation had no effect at this energy level.

25 CAL/CM²

System 1, three-ply laminates all failed at 25 cal/cm². The system 1 damage threshold is evident in Figure 1. The panel had one flash at 25 cal/cm². The failure mechanism was interlaminar face sheet delamination. All four-, five-, and six-ply laminates had sufficient mass to absorb this energy level without visual damage or structural degradation. Humidity saturation amplified the damage on a three-ply laminate. (See Figure 2.) The right-hand specimen was dry; the left-hand specimen was saturated.

30 CAL/CM²

System 1 three- and four-ply laminates all failed at 30 cal/cm^2 . The failure mechanism was interlaminar face sheet delamination. The five- and six-ply face sheets were unaffected at either one or three flashes but suffered severe paint discoloration after five flashes. (Figure 3 illustrates the difference between a three- and six-ply laminate of system 1 material at

 $^{^{\}mathrm{a}}$ All energies referenced are absorbed energies, not fluent energies.

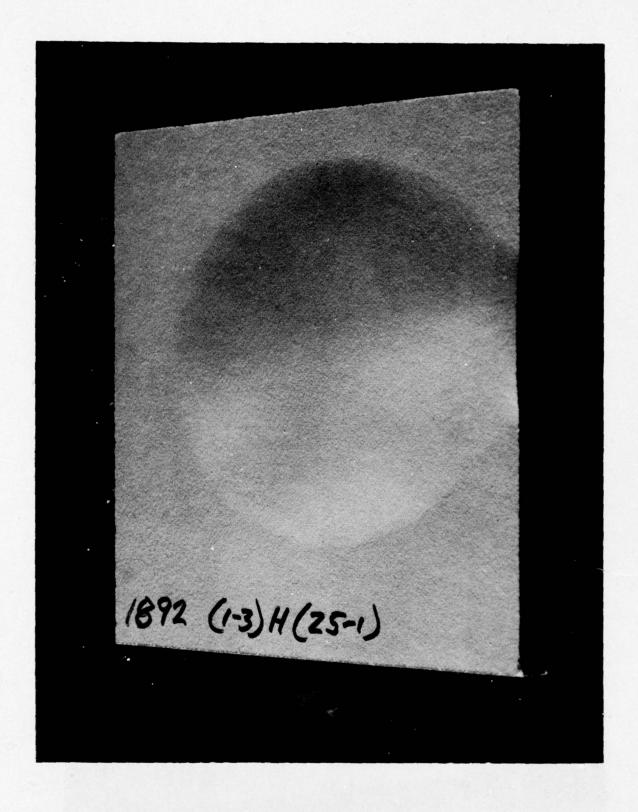


Figure 1. System 1 - material with threshold damage.

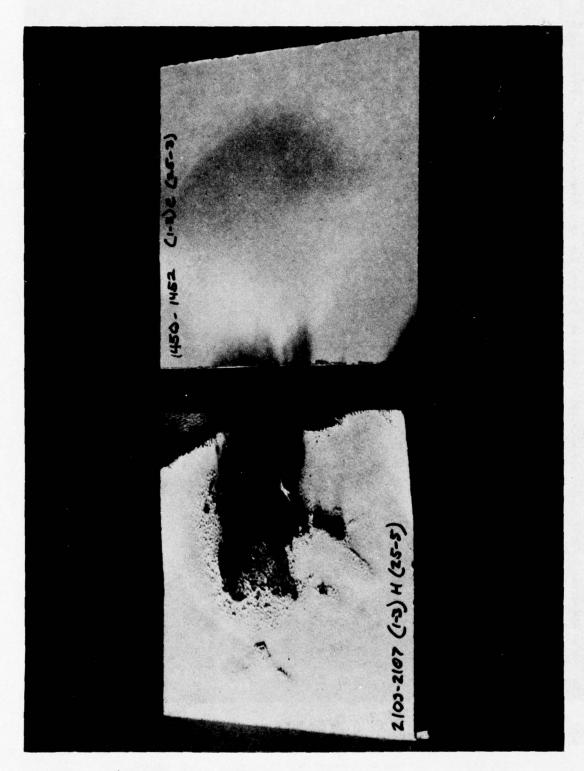


Figure 2. Humidity saturation vs dry specimen.

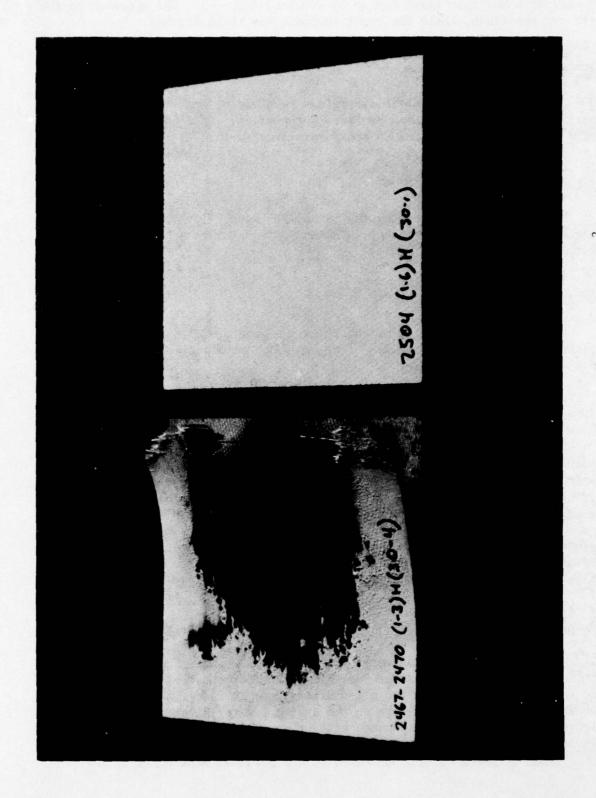


Figure 3. System 1 - 3- vs 6-ply at 30 cal/cm².

30 cal/cm².) Multiple flash damage is shown in Figure 4. The specimen on the left saw one flash, while the right specimen saw three flashes.

SYSTEM 2

System 2 was a 7781 style fiberglass-reinforced, epoxy-novalac, resinmatrix composite manufactured by Ferro Corporation. This material is sold under the trade name of CE-9000 epoxy reinforced with 7781 fiberglass.

15 CAL/CM²

System 2 was unaffected by the 15 cal/cm² energy level at any structural thickness and up to five flashes. The humidity saturation had no effect at this energy level.

20 CAL/CM²

System 2 was unaffected by the 20 cal/cm^2 energy level at any structural thickness and up to five flashes. The humidity saturation had no effect at this energy level.

25 CAL/CM²

System 2 three-, four-, and five-ply laminates all failed at 25 cal/cm² after only one flash. The failure mechanism was blistering and yellowing of the paint after one shot and a severe interlaminar delamination after two or more flashes. Figure 5 shows a system 2 laminate after three flashes at 25 cal/cm². The six-ply laminates were unaffected by up to five flashes in a dry condition. In a humidity saturated condition, the six-ply laminates were unaffected by one flash but suffered paint discoloration after three flashes and severe paint discoloration and blistering after five flashes. (Figure 6 illustrates the difference between three and five flashes.)

30 CAL/CM²

All of the system 2 laminates failed at the 30 cal/cm² energy level. The failure mechanism was severe delamination after one flash and charring and burning through one or two plies after three to five flashes. Humidity saturation amplified the damage on the system 2 material at 30 cal/cm². Figure 7 shows a five-ply specimen after three flashes. The left specimen was humidity saturated, while the right specimen was dry.

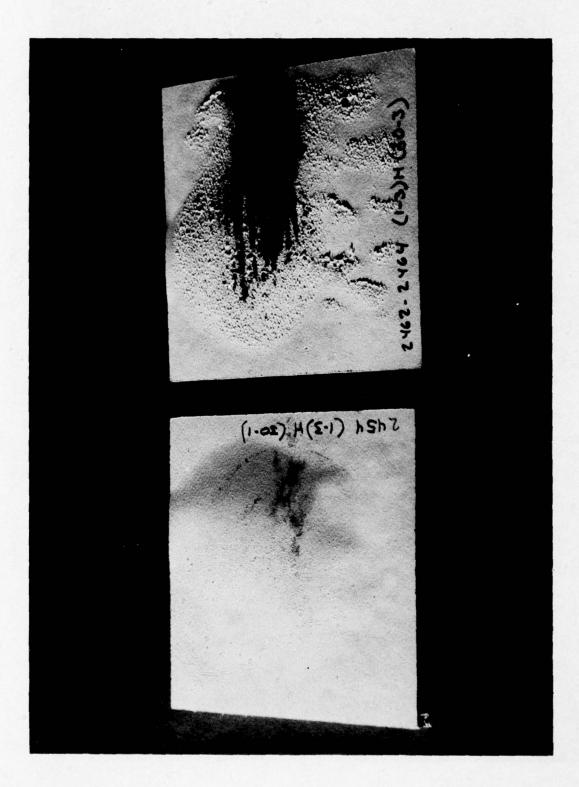


Figure 4. System 1 - one vs three flashes.



Figure 5. System 2 - three flashes at 25 cal/cm^2 .

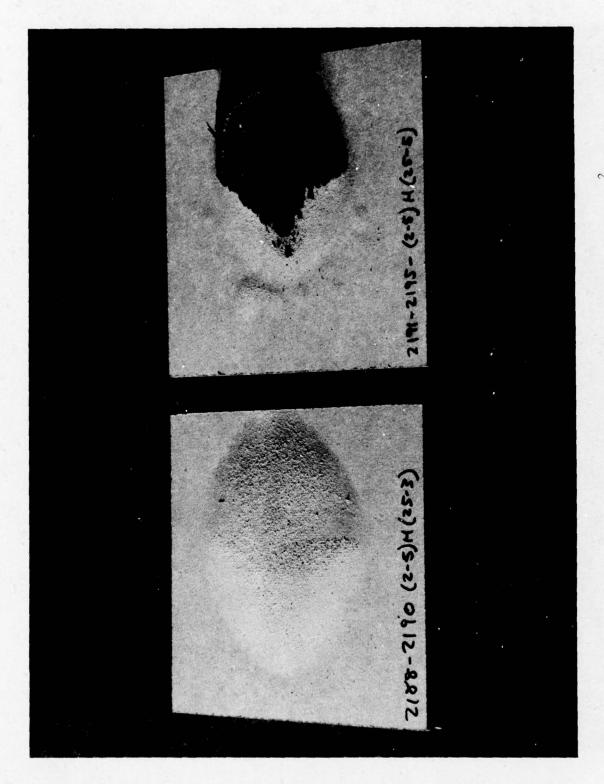


Figure 6. System 2 - three vs five flashes at 25 cal/cm^2 .

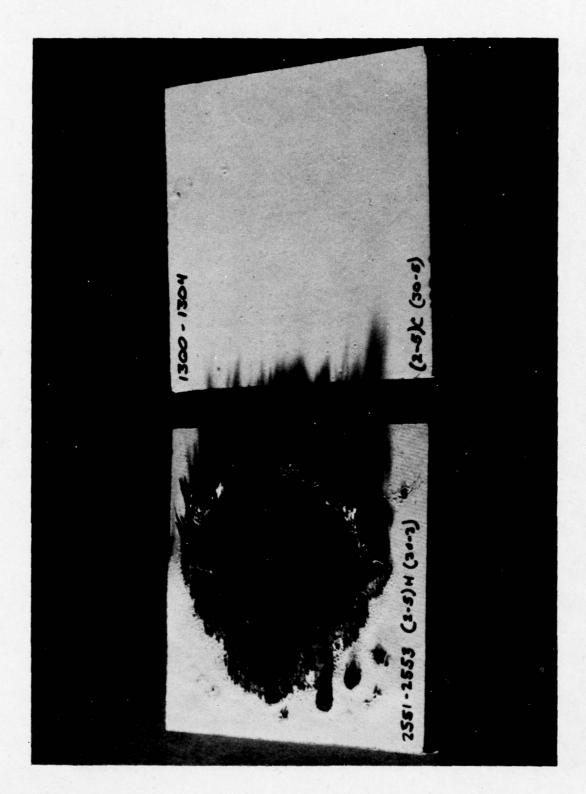


Figure 7. System 2 - saturated vs dry.

SYSTEM 3

System 3 was a 7781 style fiberglass-reinforced additional polyimide resin-matrix composites manufactured by the Hexcel Corporation. This material is sold under the Hexcel trade name of F-178 Polyimide with 7781 fiberglass reinforcement.

15 CAL/CM²

System 3 was unaffected by the 15 cal/cm^2 energy level at any structural thickness and up to five flashes. The humidity saturation had no effect at this energy level.

20 CAL/CM²

System 3 dry laminates were unaffected at any structural thickness and up to five flashes at the 20 cal/cm² energy level. After humidity saturation, the system 3 composite suffered paint blistering and discoloration at all structural thicknesses, even after one flash. After each subsequent flash, the discoloration and blistering became more severe.

25 CAL/CM²

System 3 dry laminates were unaffected at any structural thickness and up to five flashes at the 25 cal/cm^2 energy level. After humidity saturation, three-ply laminates severely delaminated after only one flash at the 25 cal/cm^2 energy level. After humidity saturation, the three-ply laminates severely delaminated after only one flash at the 25 cal/cm^2 energy level.

The saturated four-ply laminates suffered severe paint degradation with blisters and discoloration. This surface degradation began with the first flash and progressed until the fifth flash, when laminate delamination occurred. The saturated five- and six-ply laminates suffered only paint degradation through five flashes with no structural degradation.

30 CAL/CM²

System 3 dry laminates at a structural thickness of three plies delaminated severely after one flash. The dry four- and five-ply laminates were blistered and discolored after one flash at the 30 cal/cm² energy level. Each subsequent flash further degraded the coating but did not delaminate the composite. The dry six-ply laminate was unaffected after one flash, but

began to discolor after three flashes and blistered after five flashes. The humidity-saturated, three-ply laminates delaminated the same as the dry specimens, with some delamination being so violent that the face sheet was blown completely off the substrate. This saturation effect is evident in Figure 8, where the saturated specimen on the left delaminated after one flash, while the dry specimen on the right suffered only paint damage. The saturated four-and five-ply laminates exhibited paint discoloration after one flash but severely delaminated after three flashes. The humidity saturated six-ply laminate severely delaminated at the core-to-face-sheet interface after only one flash.

SYSTEM 4

System 4 was a 7781 style fiberglass-reinforced, additional polyimide resin-matrix composite manufactured by Ferro Corporation. This material is sold under the Ferro trade name of 2272 polyimide with 7781 fiberglass reinforcement.

15 CAL/CM²

System 4 dry laminates were unaffected by the 15 cal/cm² energy level at any structural thickness and up to five flashes. The humidity-saturated three- and four-ply laminates were unaffected after one flash at 15 cal/cm² but began to discolor and blister after three flashes. The discoloration and blistering continued to grow worse through five flashes, but no structural damage was visually evident. The humidity-saturated five- and six-ply laminates were unaffected up to five flashes at the 15 cal/cm² energy level.

20 CAL/CM²

System 4 dry laminates were unaffected by the 20 cal/cm² energy level at any structural thickness and up to five flashes. All of the humidity-saturated system 4 laminates suffered severe paint degradation at the 20 cal/cm² energy level. The coating failure mechanism was severe discoloration, blistering, and charring of the coating. The laminates did not delaminate either wet or dry at this energy level.

25 CAL/CM²

System 4 dry laminates were unaffected by the 25 cal/cm 2 energy level at any structural thickness and up to five flashes. The humidity-saturated three- and four-ply laminates severely delaminated after only one flash at the 25 cal/cm 2 energy level. The humidity-saturated five- and six-ply

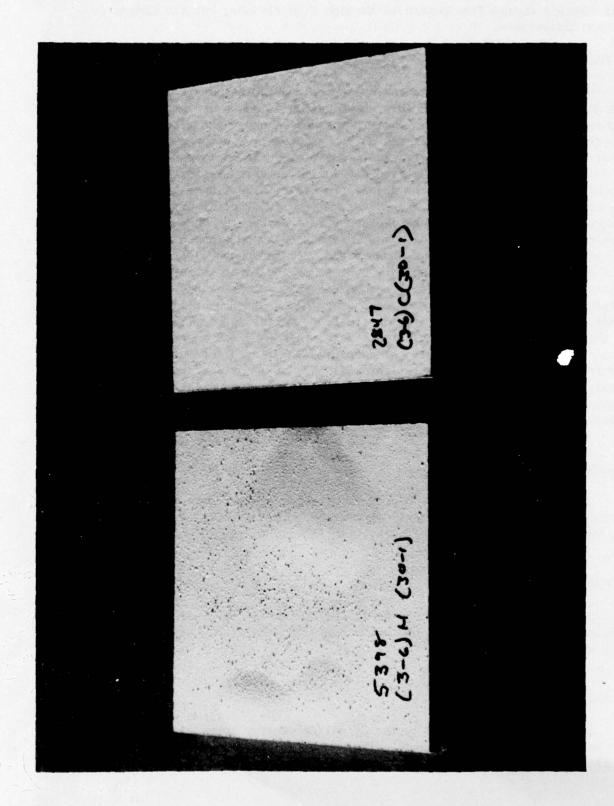


Figure 8. System 3 - humidity saturation vs dry.

laminates suffered severe coating discoloration and blistering after one flash. This coating digradation continued through five flashes, but the laminates did not delaminate.

30 CAL/CM²

System 4 dry three-ply laminates severely delaminated after one flash at the 30 cal/cm² energy level. This delamination is shown as left specimen in Figure 9. The right specimen is six plies and is undamaged after five flashes. The dry four-ply laminate suffered paint discoloration after one flash. The paint degradation became progressively worse over the next four flashes, but no delamination occurred. The five-ply laminates were unaffected by three flashes but suffered some paint degradation after five flashes. The six-ply laminates were unaffected by the 30 cal cm2 energy level at any structural thickness and up to five flashes. The humidity-saturated three-, four-, and five-ply laminates severely delaminated after only one flash at the 30 cal/cm² energy level. Figure 10 shows a saturated specimen on the left which delaminated after one flash, while the dry control on the right suffered paint discoloration after five flashes. The humidity-saturated six-ply laminate was marginal. One out of three specimens delaminated after one flash, with the other two suffering severe paint degradation. After three flashes, two out of three specimens were delaminated and the third had severe paint degradation. After five flashes, all three specimens suffered severe delamination. Figure 11 shows the damage after three flashes at 30 cal/cm² versus the damage after five flashes. One can readily see that after three flashes there is paint discoloration and blistering but no delamination; after five flashes there is severe charring and delamination of the laminate.

SYSTEM 5

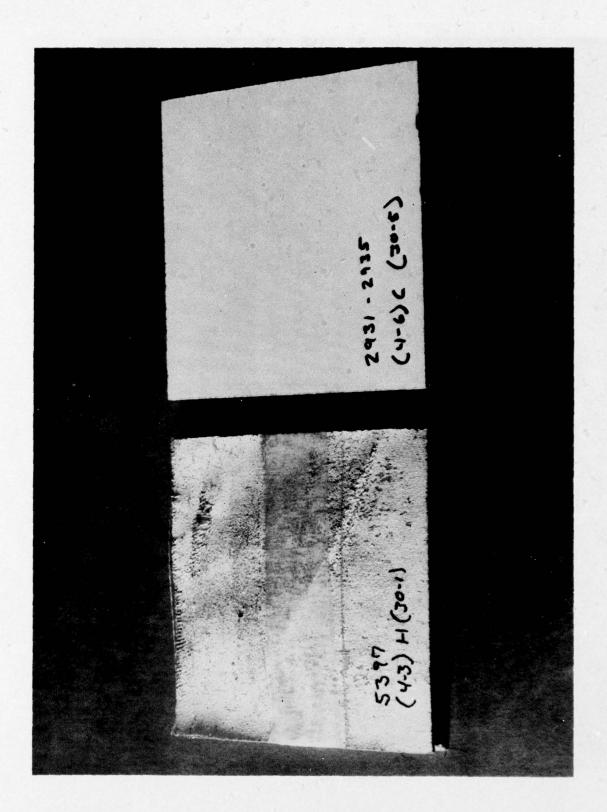
System 5 was a 581 style quartz-reinforced, epoxy-novalac, resin-matrix composite manufactured by Hexcel Corporation. This material is sold under the Hexcel trade name of F-161 epoxy with 581 quartz reinforcement.

15 CAL/CM²

System 5 was unaffected by the 15 cal/cm^2 energy level at any structural thickness and up to five flashes. The humidity saturation had no effect at this energy level.

20 CAL/CM²

System 5 was unaffected by the 20 cal/cm² energy level at any structural thickness and up to five flashes. The humidity saturation had no effect at this energy level.



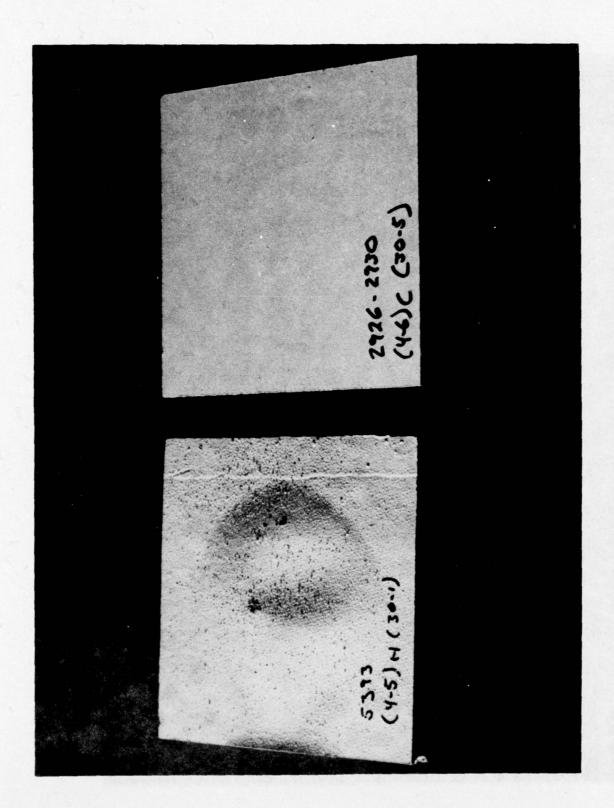


Figure 10. System 4 - humidity saturated vs dry specimens.

Figure 11. System 4 - five vs three flashes at 30 cal/cm^2 .

25 CAL/CM²

System 5 was unaffected by the 25 cal/cm² energy level at any structural thickness and up to five flashes. The humidity saturation had no effect at this energy level.

30 CAL/CM²

System 5 dry, three-ply laminates suffered minor paint discoloration at the 30 cal/cm² energy level. After each subsequent flash, the paint discoloration became more pronounced. After five flashes, the paint was light tan in color but no blistering or charring had occurred. The dry four-ply laminate was unaffected in one out of three samples after one flash. After three flashes, all specimens suffered some paint discoloration. After five flashes, the specimens had uniformally discolored but no delamination had occurred. The dry five- and six-ply laminates were unaffected by one flash. After three flashes, some coating degradation was evident; after five flashes, the samples had all evenly discolored. No coating blistering or laminate delamination was evident. The humidity-saturated three- and four-ply laminates severely delaminated after one flash at the 30 cal/cm² energy level. The fiveply laminate suffered coating discoloration at the one- and three-flash point. After five flashes, one laminate delaminated, while the other two laminates suffered severe paint degradation with charring and blistering evident. The humidity-saturated six-ply laminate was unaffected after one flash but suffered some paint discoloration after three and five flashes at the 30 cal/cm2 energy level. Figure 12 shows the initial paint blistering which occurred on system 5 after three flashes at 30 cal/cm². Figure 13 shows the coating after one flash at 30 cal/cm² on the right, and five flashes at 25 cal/cm² on the left. (Note the retention of the gloss and reflectivity even after exposure at these high levels.)

SYSTEM 6

System 6 was a 581 style quartz-reinforced, additional polyimide resinmatrix composite manufactured by Hexcel Corporation. This material is sold under the Hexcel trade name of F-178 Polyimide with 581 quartz reinforcement.

15 CAL/CM²

System 6 was unaffected by the 15 cal/cm^2 energy level at any structural thickness and up to five flashes. The humidity saturation had no effect at this energy level.

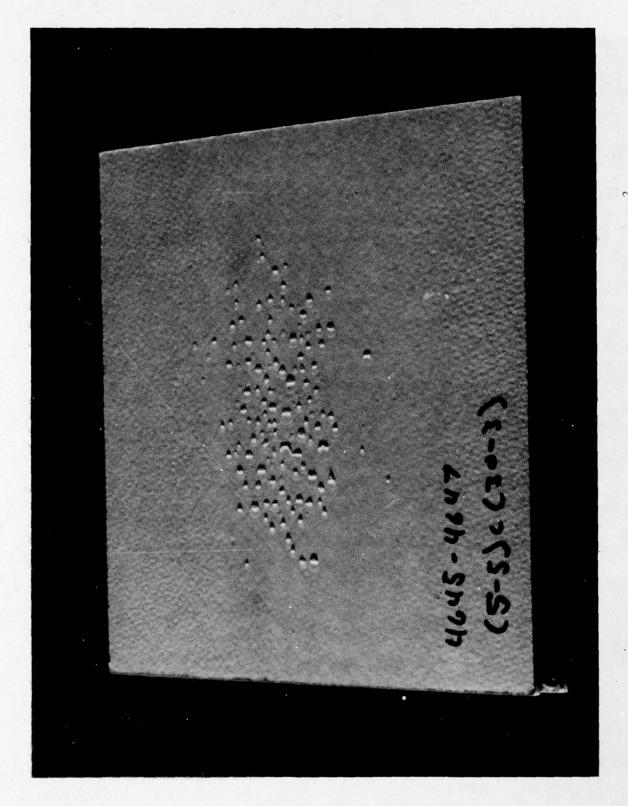


Figure 12. System 4 - paint blistering at 30 cal/cm².

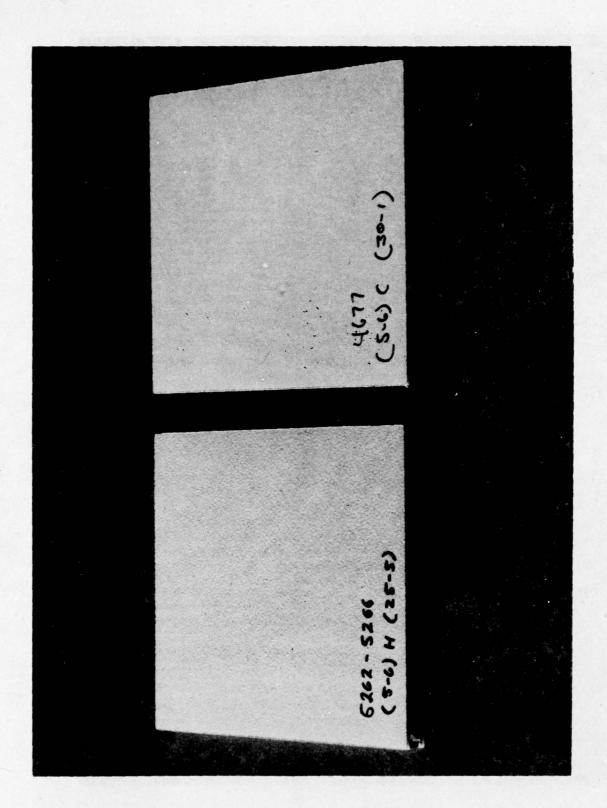


Figure 15. System 5 - coating after 25 and 30 cal/cm² exposure.

System 6 was unaffected by the 20 cal/cm² energy level at any structural thickness and up to five flashes. The humidity saturation had no effect at this energy level.

25 CAL/CM²

System 6 was unaffected after either one or three flashes at any structural thickness. After five flashes, all structural thicknesses suffered paint discoloration with the three-ply specimen showing some signs of blistering. The humidity saturation did not affect the specimen adversely at the 25 cal/cm² level. The results were the same as those obtained on the dry samples.

30 CAL/CM²

System 6 dry composites suffered paint discoloration and blistering at all structural thicknesses at the 30 cal/cm² energy level. (See Figure 14.) The paint degradation became progressively worse through five flashes, but no composite delamination occurred. The humidity-saturated composite severely delaminated after one flash at all structural thicknesses when exposed to the 30 cal/cm² energy level. This damage can be seen in Figure 15. The sample on the right was dry, while the left sample was saturated. The blistering and delamination is readily visible in the saturated specimen.

SYSTEM 7

System 7 was a T300 graphite-reinforced, epoxy-novalac, resin-matrix composite manufactured by the Celanese Corporation. This material is sold under the Celanese trade name of 5208 epoxy reinforced with T-300 graphite.

15 CAL/CM²

System 7 dry three-ply material suffered paint discoloration at the 15 cal/cm² energy level after only one flash. After each subsequent flash, the discoloration became more pronounced. The four-, five-, and six-ply dry laminates were unaffected by the 15 cal/cm² energy level. After humidity saturation, all of the system 7 composites suffered paint degradation after only one flash regardless of structural thickness. The paint degradation became more pronounced with each flash through five, but no composite delamination occurred.

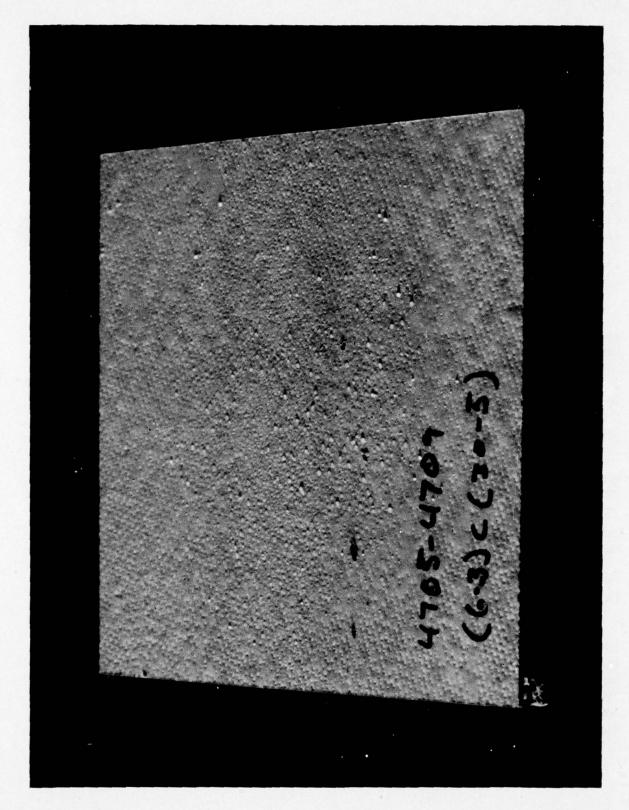


Figure 14. System 6 - threshold damage.

Figure 15. System 6 - humidity saturation vs dry specimens.

System 7 three-ply material, both dry and humidity-saturated, suffered severe coating and laminate surface charring and delamination after only one flash. Figure 16 shows the one flash damage to a three-ply laminate at 20 cal/cm². The four-, five-, and six-ply laminates, both dry and humidity-saturated, suffered paint degradation after one flash. After each subsequent flash, the degradation became more severe, with blistering and charring after five flashes.

25 CAL/CM²

System 7 dry and humidity saturated, 3- and 4-ply composite suffered severe coating and composite charring after one flash at the 25 cal/cm² energy level. The dry, 5- and 6-ply material suffered only paint degradation after up to five flashes at the 25 cal/cm² energy level. The humidity saturated 5- and 6-ply laminates suffered some surface charring of the resin matrix and some removal of the reflective coating after only one flash. Figure 17 is a comparison of a dry 6-ply laminate after one flash at 25 cal/cm², and a saturated 6-ply laminate tested at the same level. Note the increased paint damage in the saturated specimen. Figure 18 illustrates the effect of multiple flashes on a five-ply laminate at 25 cal/cm². After one flash, the right specimen has coating damage; after five flashes, the left specimen shows charring of the laminate.

30 CAL/CM²

The dry three-, four-, and five ply laminates suffered severe charring of the surface resin after only one flash at the 30 cal/cm² energy level. The dry six-ply laminate suffered only paint discoloration after one flash but proceeded to charr and degrade the laminate on each subsequent flash. All system 7 humidity-saturated composites were severely degraded after only one flash, regardless of thickness, at the 30 cal/cm² energy level.

SYSTEM 8

System 8 was an AS graphite-reinforced, epoxy-novalac, resin-matrix composite manufactured by the Hercules Corporation. This material is sold under the Hercules trade name of 3501-5A epoxy with AS graphite reinforcement.

Figure 16. System 7 - damage to 3-ply specimen at 20 cal/cm².

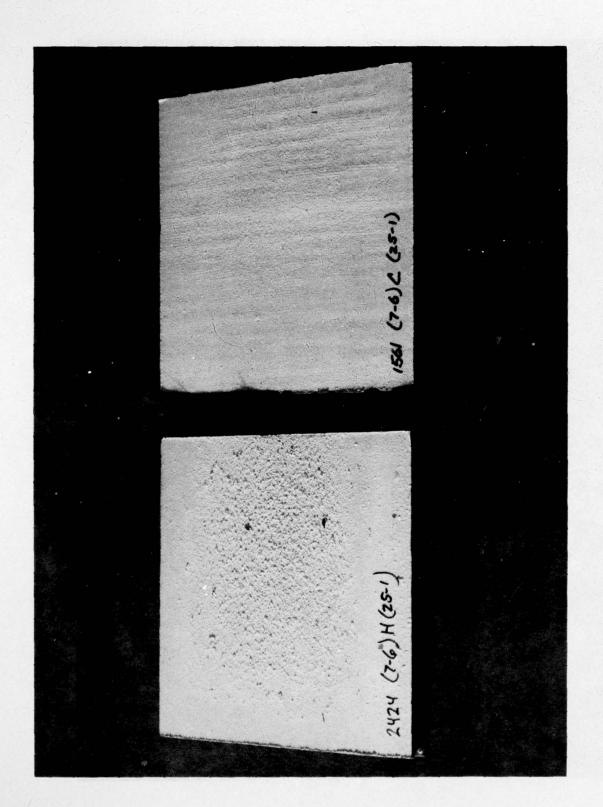


Figure 17. System 7 - humidity saturated as dry specimen at 25 cal/cm 2 .

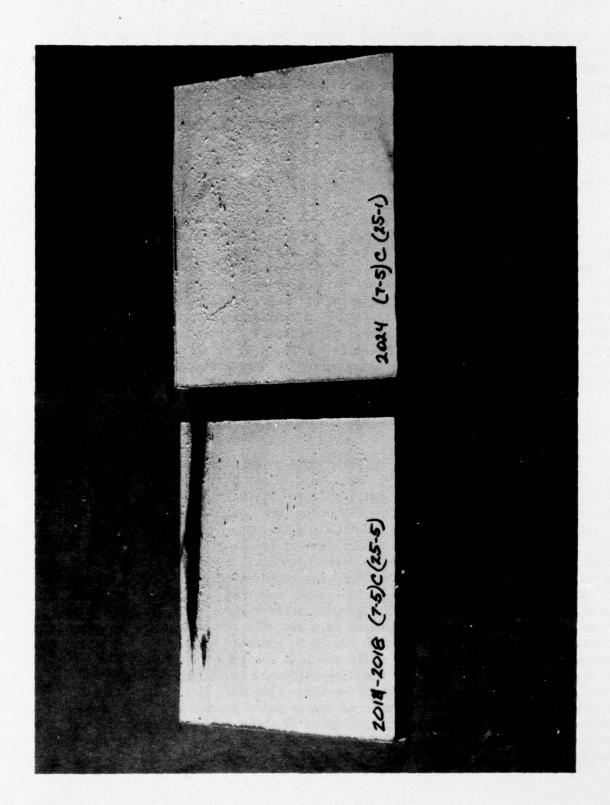


Figure 18. System 7 - one flash vs five flashes at 25 $\operatorname{cal/cm}^2$.

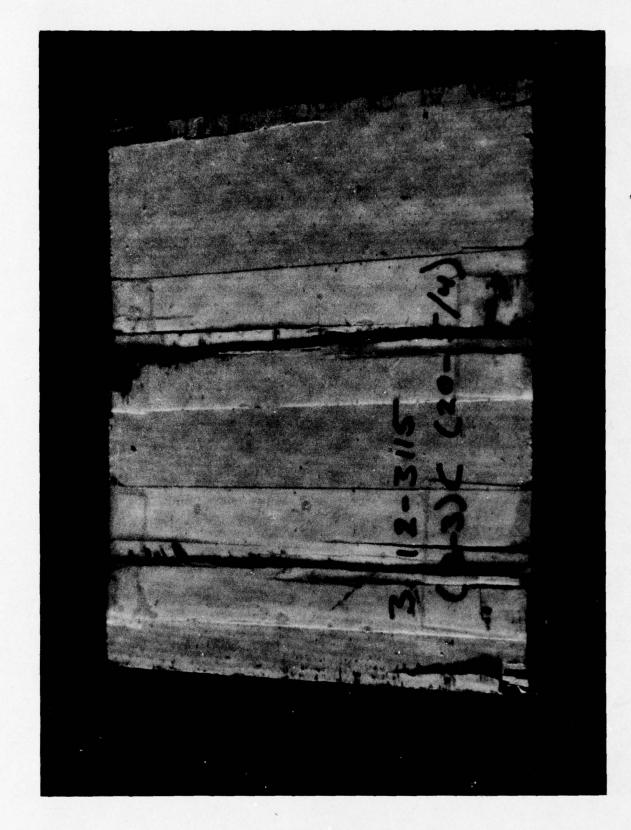
System 8,dry three-, four-, and five-ply laminates suffered paint discoloration after one flash and some paint blistering after three and five flashes. No laminate delamination or charring occurred at the 15 cal/cm² energy level. The dry six-ply laminates were unaffected up to five flashes at the 15 cal/cm² energy level. All of the humidity-saturated laminates suffered paint discoloration after one flash and blistering and charring after three and five flashes.

20 CAL/CM²

The dry three-ply laminate suffered paint discoloration after one flash, paint blistering and charring after three flashes, and severe resin-matrix degradation and laminate delamination after four flashes. This degradation is shown in Figure 19. The dry four-, five-, and six-ply laminates suffered paint discoloration after one flash. This paint degradation progressed after each flash to blisters and charring after five flashes at the 20 cal/cm energy level. All of the humidity-saturated laminates suffered paint discoloration after one flash, regardless of thickness. After each subsequent flash, the paint degradation became more severe, with blisters and charring after five flashes. No delamination occurred at this energy level on the humidity saturated laminates.

25 CAL/CM²

The dry system 8 composites suffered only paint discoloration after one flash at the 25 ca1/cm² energy level, regardless of thickness. After three flashes, the three-ply laminates suffered severe resin-matrix charring and complete removal of the coating. The dry four-, five-, and six-ply laminates suffered some paint degradation and laminate charring after the three and five flashes at the 25 cal/cm² energy level. Figure 20 illustrates the progressive damage from three to five flashes at 25 cal/cm2. The threeply humidity-saturated laminate suffered severe resin-charring matrix after only one flash. The four-, five-, and six-ply specimen suffered only paint degradation after one flash at 25 cal/cm2. After three flashes, the humiditysaturated four- and five-ply laminates were severely resin charred. Figure 21 illustrates the damage amplification caused by humidity saturation. The right specimen shows paint damage dry, while the left specimen was saturated and had resin charring. The six-ply humidity-saturated laminate suffered only paint discoloration and blistering after three flashes. After five flashes, the saturated six-ply laminate suffered severe paint degradation with extensive resin charring of the laminate. Figure 22 shows the comparative degradation between three- and six-ply dry laminate at 25 cal/cm2. (Note the resin charring on the three-ply but only paint damage at six-plies.)



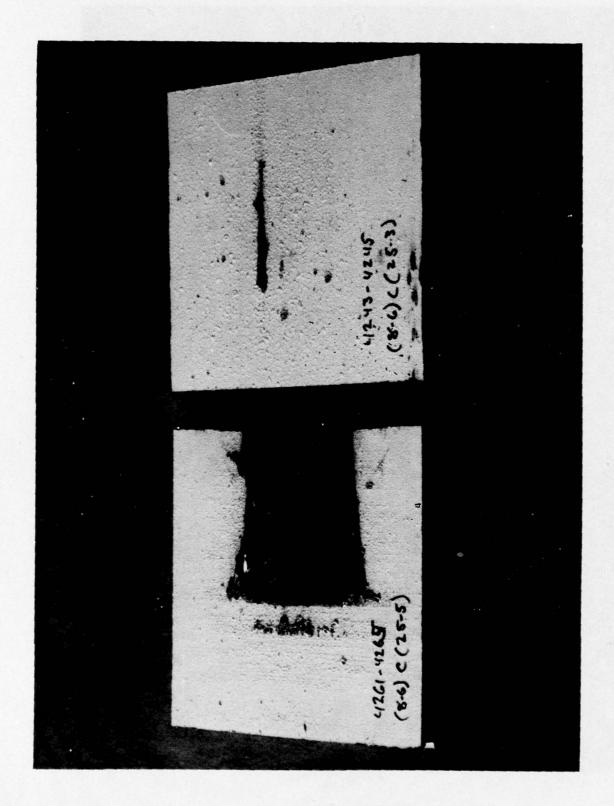


Figure 20. System 8 - progressive damage, three to five flashes at 25 cal/cm².

Figure 21. System 8 - humidity saturated vs dry specimen at 25 cal/cm².

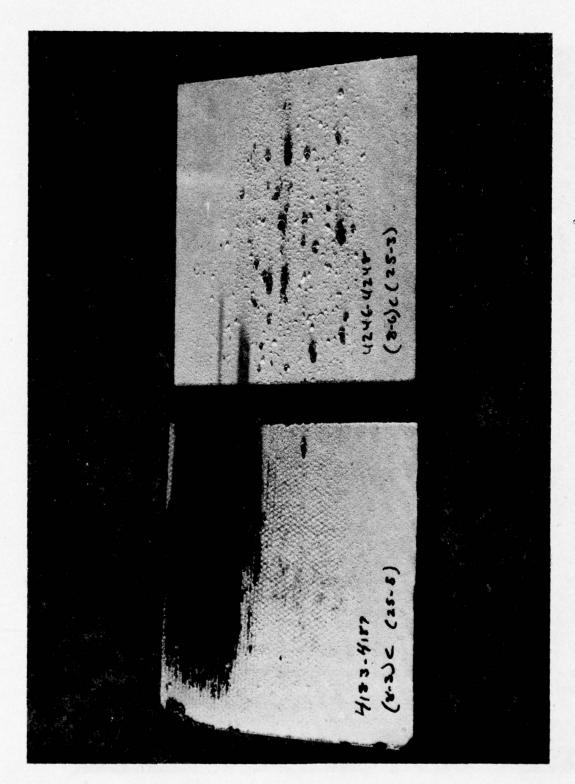


Figure 22. System 8 - 3 vs 6 ply at 25 cal/cm^2 .

System 8 composite laminates suffered severe resin-matrix burnout after only one flash in either a dry or humidity-saturated condition.

SYSTEM 9

System 9 was an AS graphite-reinforced, epoxy-novalac, resin-matrix composite manufactured by the Fiberite Corporation. This material is sold under the Fiberite trade name of 934 epoxy with AS graphite reinforcement.

15 CAL/CM²

System 9 was unaffected by one flash at the 15 cal/cm² energy level at any structural thickness when dry. After three flashes, the dry three-ply laminate was discolored. The dry four-, five-, and six-ply laminates were unaffected after three flashes at the 15 cal/cm² energy level. After five flashes the three-, four-, and five-ply dry laminates were discolored. The dry six-ply laminate was unaffected by five flashes at the 15 cal/cm² energy level. After humidity saturation, all system 9 laminates suffered paint discoloration and blistering after one flash. Each subsequent flash caused further paint degradation but no laminate charring or delamination. (See Figure 23.)

20 CAL/CM²

All system 9 composite samples suffered paint discoloration and blistering at the 20 cal/cm² energy level. The thinner laminates had more degradation than the six plies, but all suffered considerable paint loss. The humidity-saturated material had more paint blisters than the dry, but none of the panels delaminated or charred.

25 CAL/CM²

System 9 dry laminates suffered only paint degradation after one flash, regardless of thickness. After three flashes, all thicknesses of dry composite suffered severe laminate degradation, with extensive resin burnout evident. Figure 24 illustrates the progressive delamination which occurred between three and five flashes at 25 cal/cm². The humidity-saturated three-and four-ply laminates suffered severe laminate damage after one flash. The five- and six-ply saturated laminates suffered only paint discoloration and blistering after one flash. After three flashes, the saturated laminates suffered severe resin charring and fiber loss.

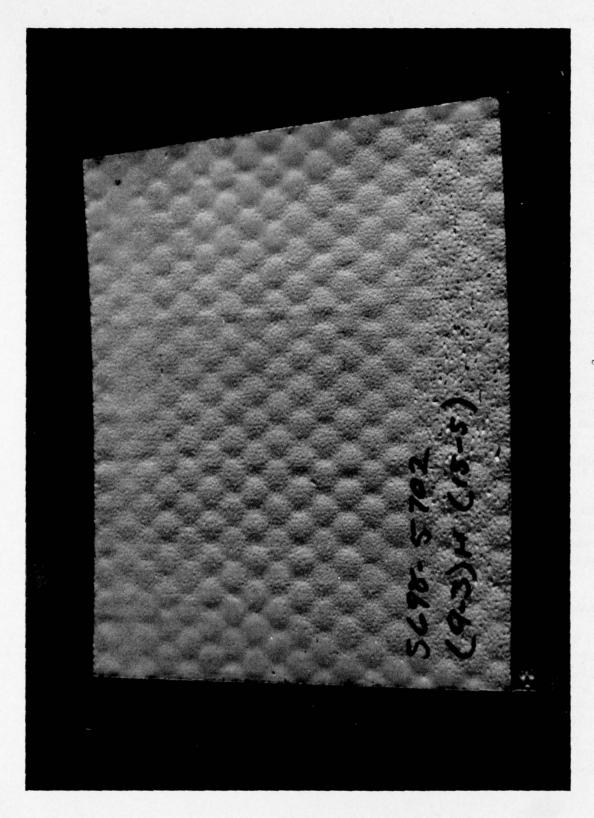


Figure 23. System 9 - 15 cal/cm² paint degradation.

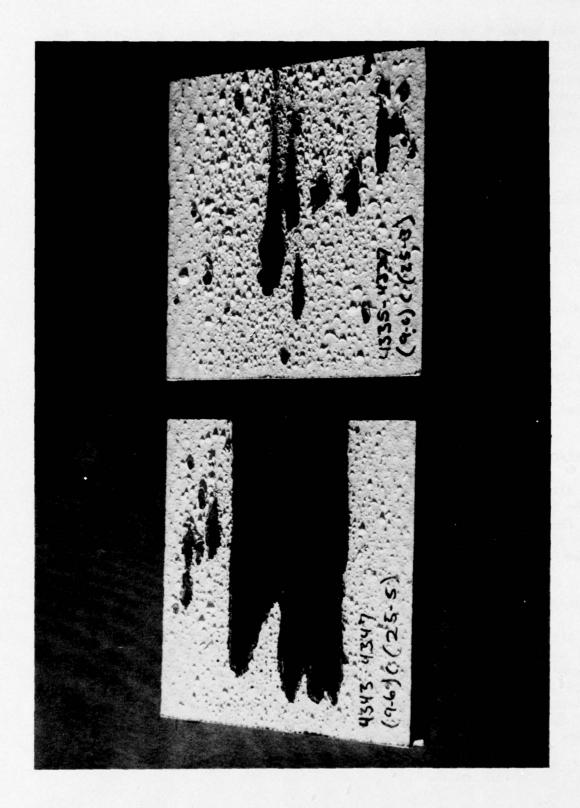


Figure 24. System 9 - progressive degradation from three to five flashes at 25 cal/cm².

All system 9 laminates suffered extensive damage after one flash at the 30 cal/cm² energy level. The failure mechanism was charring and resin burnout from the surface. At this energy level, the dry and humidity-saturated laminates reacted in the same manner. Figure 25 illustrates the difference in laminate degradation caused by reductions in structural thickness. The specimen on the right is six plies thick and suffered moderate laminate charring after one flash at 30 cal/cm². The specimen on the left was three plies thick and suffered extensive resin charring after one flash at 30 cal/cm².

SYSTEM 10

System 10 was an AS graphite-reinforced, additional polyimide resin-matrix composite manufactured by Hexcel Corporation. This material is sold under the Hexcel trade name of F-178 polyimide with AS graphite reinforcement.

15 CAL/CM²

The dry system 10 laminates were unaffected at the 15 cal/cm² energy level, regardless of thickness and up to five flashes. All humidity-saturated laminates suffered paint discoloration and blistering at the 15 cal/cm² energy level, regardless of specimen thickness and up to five flashes.

20 CAL/CM²

All system 10 laminates suffered paint discoloration and blistering at the 20 cal/cm² energy level, regardless of specimen thickness and up to five flashes. The humidity-saturated laminates all suffered similar paint discoloration but had more evidence of paint blistering and flaking. None of the laminates exposed at 20 cal/cm² had any resin charring or delamination.

25 CAL/CM²

All system 10 dry specimens had paint discoloration and blistering after one flash at 25 cal/cm². The dry three-, four-, and five-ply laminates suffered resin charring and delamination after exposure to three flashes at the 25 cal/cm² energy level. Figure 26 illustrates this damage on a three-ply laminate after two flashes at 25 cal/cm². The dry six-ply laminate had only paint discoloration and blistering after three flashes. After five flashes, the dry three-, four-, and five-ply laminates had severe resin charring and



Figure 25. System 9 - 3- vs 6-ply specimen at 30 cal/cm² (one flash).

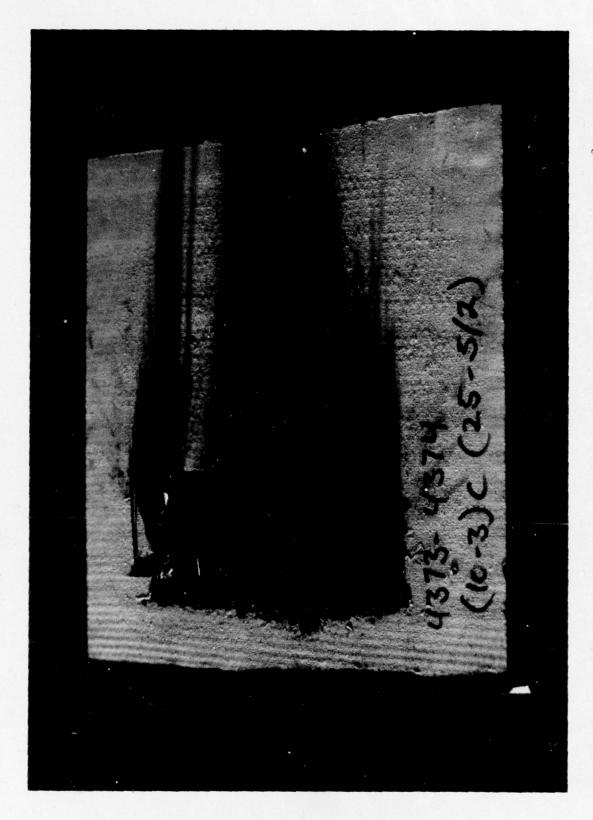


Figure 26. System 10 - specimen degradation after two flashes at 25 cal/cm².

fiber loss. The dry six-ply laminate had only paint discoloration and blistering after five flashes at 25 cal/cm². All humidity-saturated laminates suffered paint discoloration and blistering after one flash but suffered charring, resin, and fiber loss after three flashes at the 25 cal/cm² energy level.

30 CAL/CM²

All System 10 composite specimens suffered extreme resin and fiber degradation after only one flash at the $30~ca1/cm^2$ energy level. The dry and the humidity-saturated laminates reacted in the same manner at this energy level. Figure 27 illustrates the effects of mass on laminate degradation. The specimen on the left was six plies thick and suffered some resin charring and fiber loss after one flash. The right-hand specimen was only four plies thick and suffered extensive resin charring and fiber loss at the same $30~ca1/cm^2$ energy level.

SYSTEM 11

System 11 was a 181 style Kevlar-reinforced, epoxy-novalac, resin-matrix composite manufactured by the Celanese Corporation. This material is sold under the Celanese trade name of 5208 epoxy with 181 Kevlar reinforcement.

15 CAL/CM²

The system 11 dry laminates were unaffected at the 15 cal/cm² energy level. The humidity-saturated three- and four-ply laminates suffered paint discoloration after the first flash and became progressively darker with each subsequent flash through five. The humidity-saturated five- and six-ply laminates were unaffected up to five flashes at the 15 cal/cm² energy level.

20 CAL/CM²

All system 11 dry specimens suffered some discoloration after one flash. Each subsequent flash caused further paint degradation but no resin charring or delamination. The humidity-saturated specimens discolored and blistered after one flash at the 20 cal/cm² energy level. Each subsequent flash caused further blistering and discoloration of the coating through five flashes. No charring or delamination of the substrate was evident.

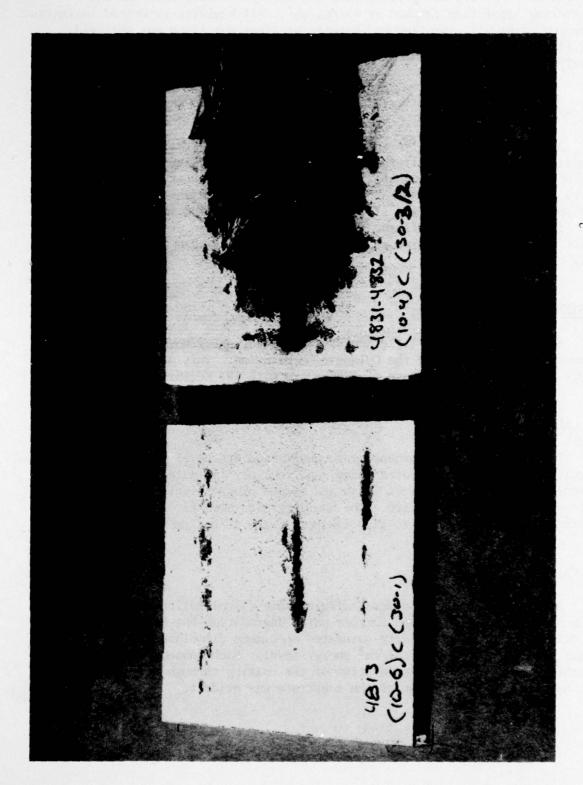


Figure 27. Four- vs 6-ply specimen at 30 cal/cm².

All system 11 dry laminates suffered paint discoloration after one flash. The humidity-saturated specimens were severely blistered and discolored after one flash at the 25 cal/cm² energy level. After three flashes, the dry specimens began to charr and burn, with two out of three specimens exhibiting some charring of the surface resin. After five flashes, all laminates suffered severe charring. (See Figure 28.) Figure 29 demonstrates the progressive nature of this damage. The five-ply laminate on the right is blistered after three flashes, while the left sample is charred after four flashes at the same energy level.

30 CAL/CM²

All system 11, dry specimens suffered paint discoloration after one flash at 30 cal/cm². After three flashes, all dry specimens suffered some paint blistering and resin burning. After five flashes, all dry specimens suffered resin charring. All system 11 humidity-saturated specimens suffered some resin charring after one flash at the 30 cal/cm² energy level. (See Figure 30.) Figure 31 illustrates the effects of laminate thickness on degradation. The specimen on the right has only paint blistering and discoloration after one flash at 30 cal/cm², while the left- three-ply specimen is severely degraded after three flashes at only 25 cal/cm².

SYSTEM 12

System 12 was a 181 style Kevlar-reinforced, epoxy-novalac, resin-matrix composite manufactured by Hexcel Corporation. This material is sold under the Hexcel trade name of F-161 epoxy with 181 Kevlar reinforcement.

15 CAL/CM²

All system 12 composite specimens were unaffected up to five flashes at the 15 cal/cm^2 energy level. Neither the specimen thickness not the humidity saturation had any effect on these specimens.

20 CAL/CM²

The system 12 specimens were unaffected up to five flashes at the 20 cal/cm² energy level. Neither the specimen thickness nor the humidity saturation had any effect on these specimens.

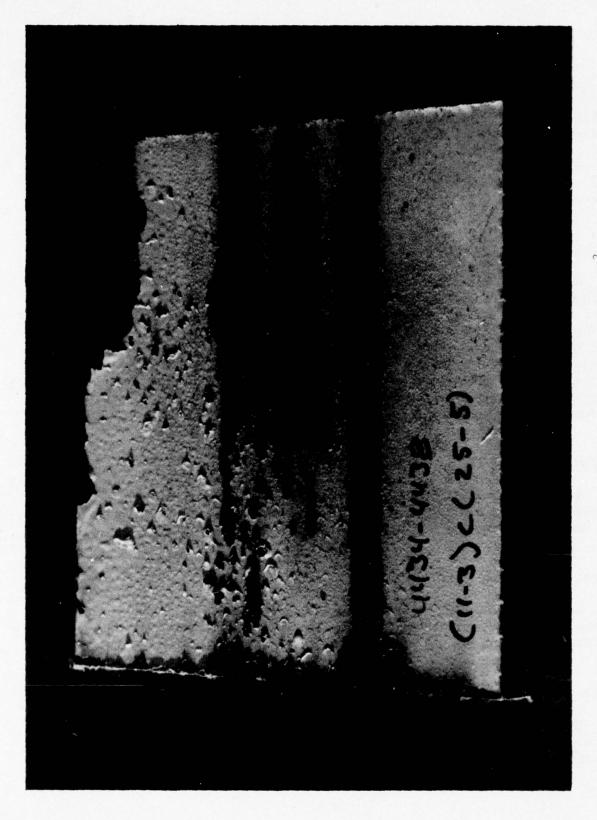


Figure 28. System 11 - specimen degradation at 25 cal/cm² (five flashes).

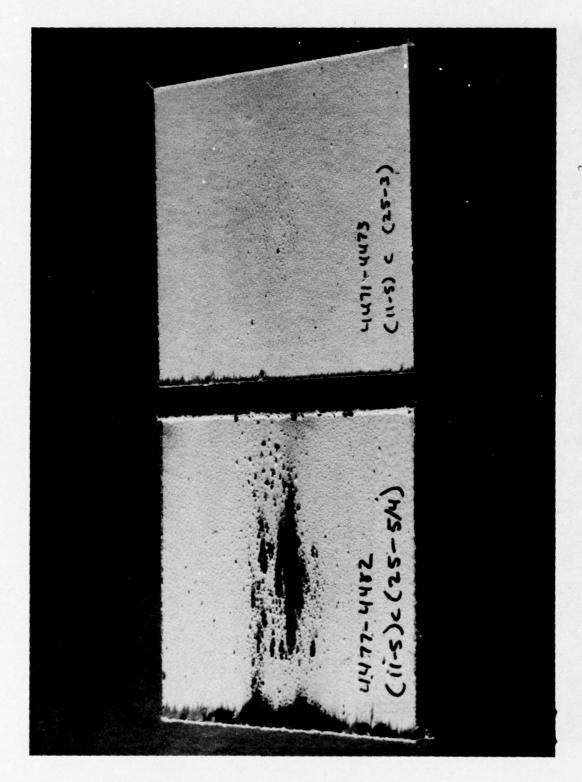


Figure 29. System 11 - three vs four flashes at 25 cal/cm².

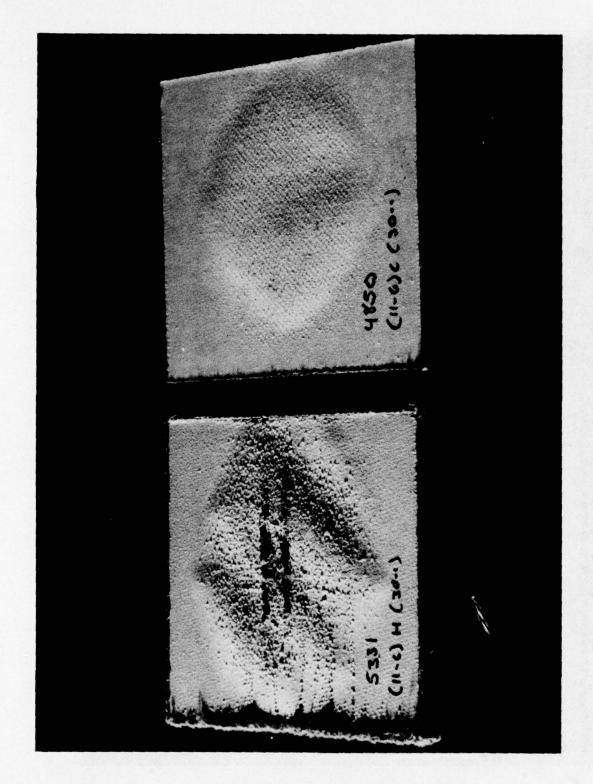


Figure 30. System 11 - resin charring and delamination of 6-ply specimen.

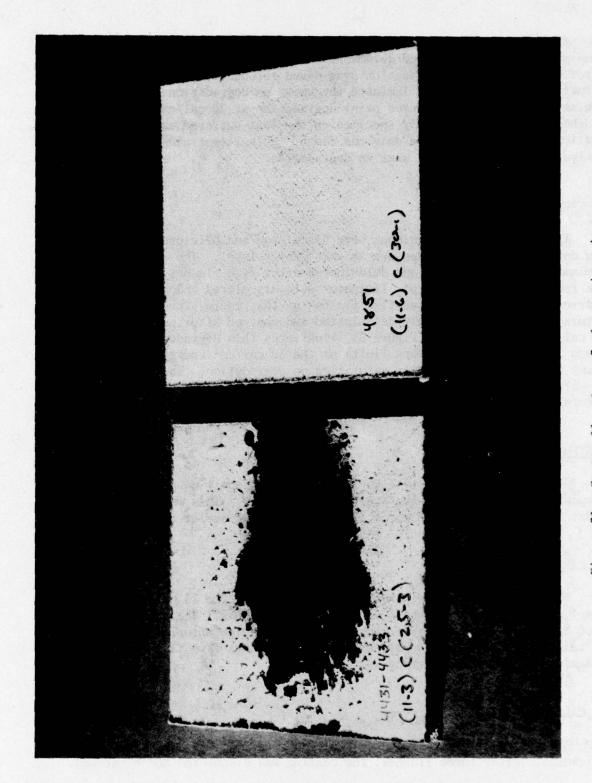


Figure 31. System 11 - 6- vs 3-ply specimen damage.

All system 12 composite specimens, both saturated and dry, suffered some paint discoloration and delamination at the 25 cal/cm² energy level. After one flash, this degradation progressed through five flashes. Figure 32 illustrates the effects of laminate thickness on degradation. The six-ply laminate on the right suffered paint degradation at 30 cal/cm² after five flashes, while the three-ply specimen on the left suffered charring and delamination at 25 cal/cm² after only one flash. This illustrates the effect of laminate thickness or heat sink on degradation.

30 CAL/CM²

The system 12 dry specimens were discolored and blistered after both the one- and five-flash exposure at this energy level. The dry three-ply laminate severely charred and delaminated after five flashes. (See Figure 33.) The four-, five-, and six-ply laminates were discolored and blistered but not charred or delaminated after five flashes at this energy level. All humidity-saturated laminates severely delaminated and charred after one flash at the 30 cal/cm² energy level. Figure 34 illustrates this degradation after one flash (right) and two flashes (left) at the 30 cal/cm² energy level. Figure 35 shows the effects of humidity saturation on degradation. The right specimen is only moderately charred dry, while the saturated specimen on the left is severely charred and delaminated. Both were tested at 30 cal/cm².

SYSTEM 13

System 13 was a 181 style Kevlar-reinforced, epoxy-novalac, resin-matrix composite manufactured by the Fiberite Corporation. This material is sold under the Fiberite trade name of 934 epoxy with 181 Kevlar reinforcement.

15 CAL/CM²

The system 13 dry laminates were unaffected by the 15 cal/cm 2 energy level, regardless of thickness and up to five flashes. The humidity-saturated three-ply laminates suffered paint discoloration after one flash at 15 cal/cm 2 . The coating continued to darken through five flashes, but no delamination or resin charring was evident.

20 CAL/CM²

All system 13 dry laminates discolored after one flash, regardless of thickness. After three flashes, the coating was discolored and blistered.

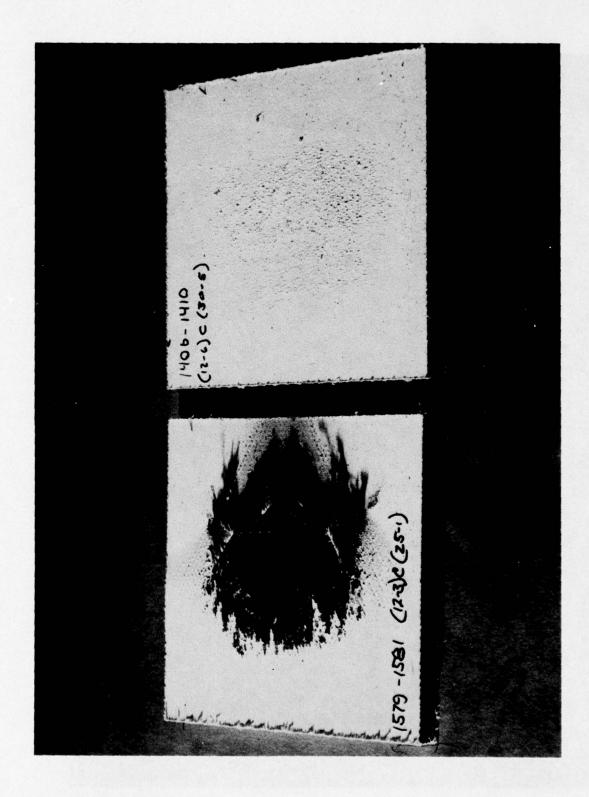


Figure 32. System 12 - 3- vs 6-ply specimen.

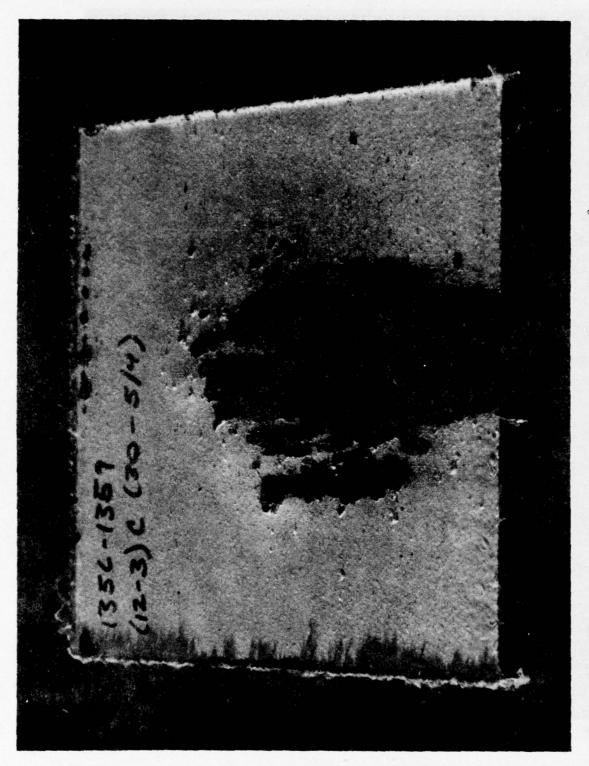


Figure 33. System 12 - damage at 30 cal/cm².

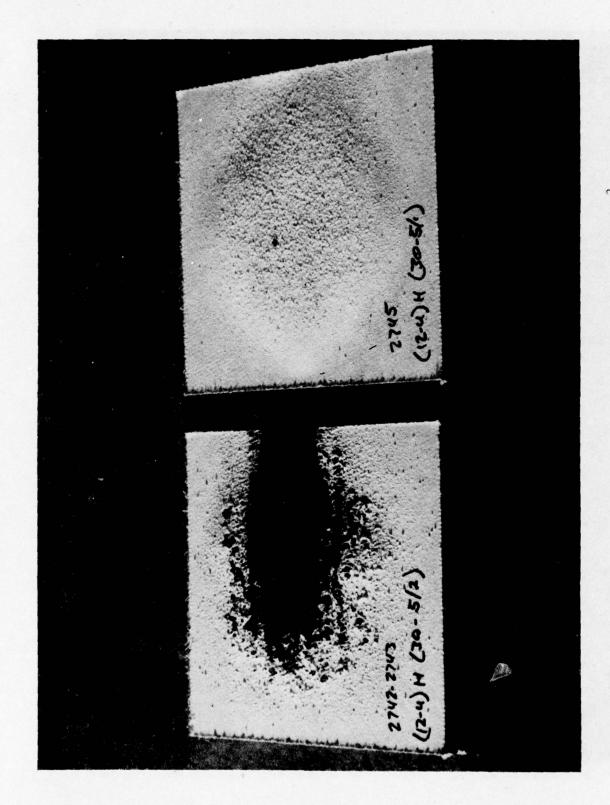


Figure 34. System 12 - one vs two flashes at 30 cal/cm².

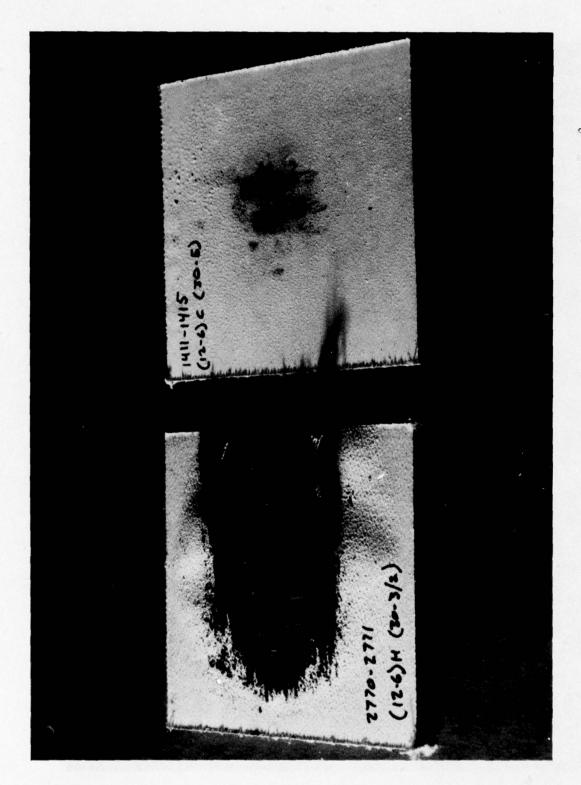


Figure 55. System 12 - humidity saturated vs dry specimen at 30 cal/cm².

The discoloration and blistering continued on the dry laminates through five flashes. The humidity-saturated three-ply laminates discolored after one flash, but one out of three disbonded and blistered after three flashes. After five flashes, two out of three of the humidity-saturated composite specimens were delaminated and charred. The four-, five-, and six-ply humidity-saturated laminates discolored after one flash but continued through five flashes, with only further paint discoloration and blistering but no delamination or charring of the resin substrate.

25 CAL/CM²

System 13 dry three-ply laminates were charred and delaminated after one flash at 25 cal/cm². (See Figure 36.) The four-, five-, and six-ply dry laminates suffered paint discoloration and blistering after one flash but did not char or delaminate. After three flashes, the dry three- and four-ply laminates charred and delaminated. The five- and six-ply dry laminates suffered only discolored paint. This thickness effect on system 13 is seen in Figure 37, where the right six-ply specimen is delaminated at 30 cal/cm², while the left three-ply specimen is charred at only 25 cal/cm². After five flashes, the three-, four-, and five-ply laminates all charred and delaminated. The right specimen is only delaminated after one flash, while the left specimen is charred after three flashes. (See Figure 38.) The six-ply laminate suffered only paint blistering and discoloration. After humidity saturation, the three-, four-, and five-ply laminates delaminated and charred after only one flash. After three flashes, all humidity-saturated laminates charred and delaminated.

30 CAL/CM²

All system 13 composite laminates, both dry and humidity-saturated, failed after one flash at the 30 cal/cm² energy level. The thin three-ply laminates suffered more charring than the six plies, but all were severely delaminated. Figure 39 contrasts the dry and saturated laminates of 30 cal/cm². The dry specimen on the right has minor delamination, while the left specimen has charring and major delamination for saturated exposure at this energy level.

SYSTEM 14

System 14 was a boron-reinforced, epoxy-novalac, resin-matrix composite manufactured by Avco Corporation. This material is sold under the Avco trade name of 5505 epoxy resin with boraon reinforcement.

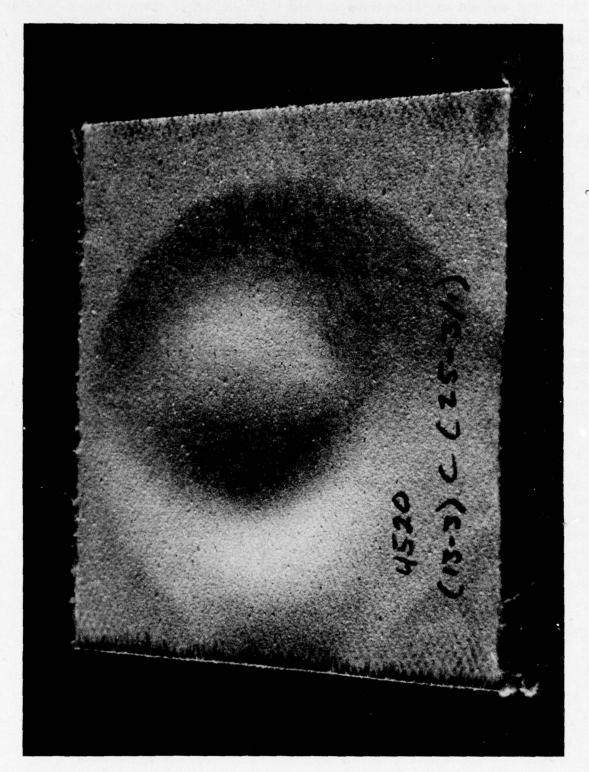


Figure 36. System 13 - delamination at 25 cal/cm².

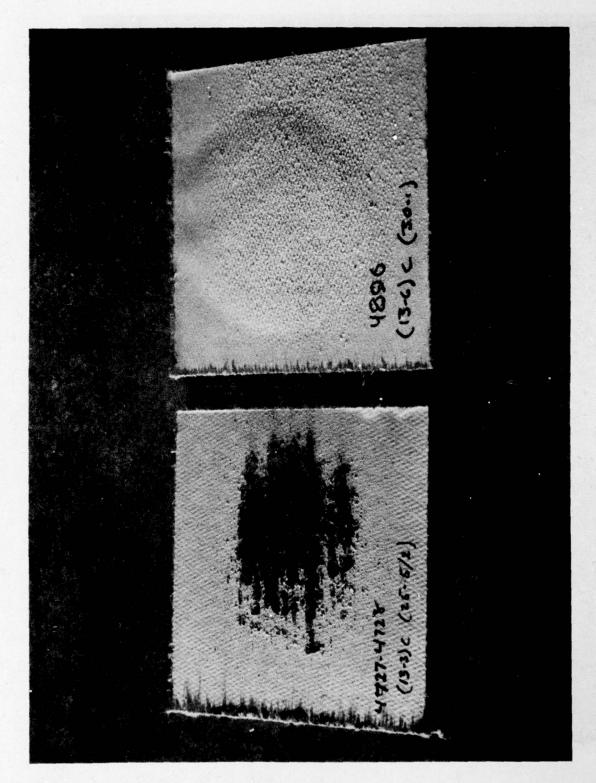


Figure 37. System 13 - 3- vs 6-ply damage.

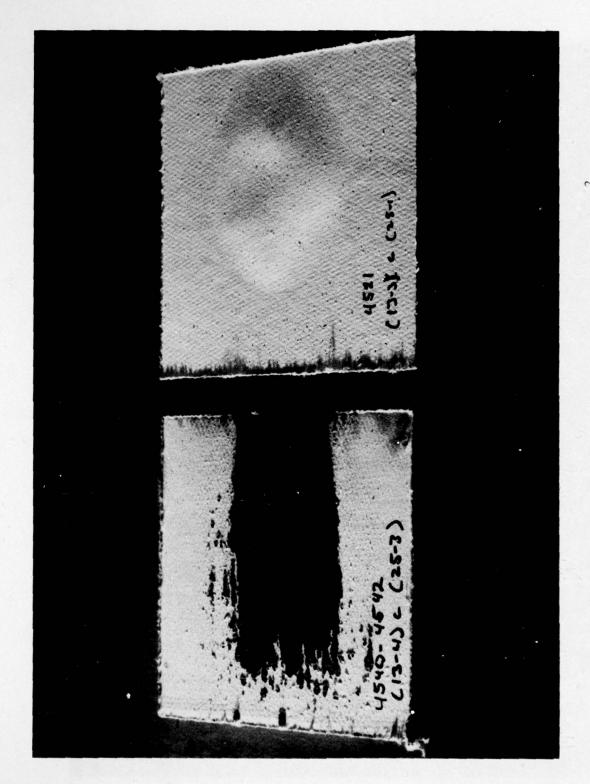


Figure 38. System 13 - one vs three flashes at 25 $\operatorname{cal/cm}^2$.

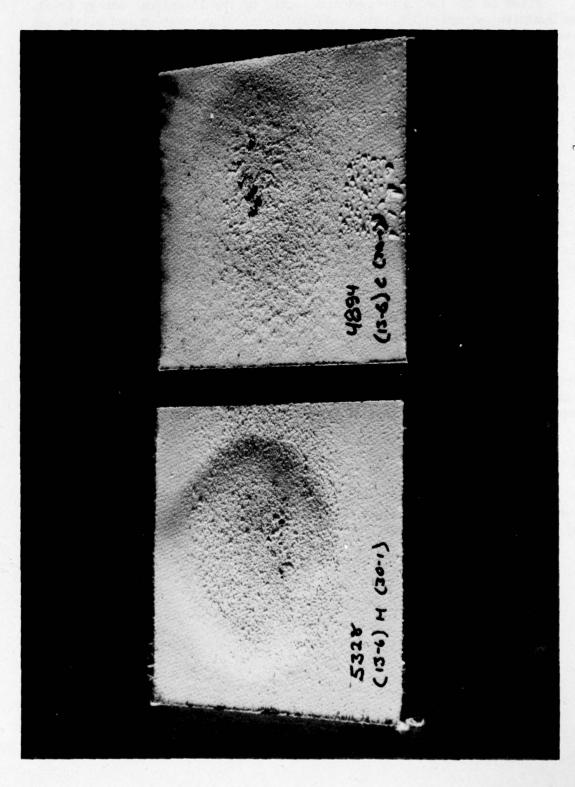


Figure 39. System 13 - humidity vs dry specimen at 30 $\operatorname{cal/cm}^2$.

System 14 dry laminates were unaffected by the 15 cal/cm^2 energy level. The humidity-saturated laminates suffered paint discoloration and blistering after one flash. The coating damage progressed with each subsequent flash. The thin laminates were degraded more than the thick, but all suffered some coating failure.

20 CAL/CM²

All system 14 composite laminates suffered severe coating and composite delamination failures at the 20 cal/cm² energy level. The thickness of the specimens had little effect on the damage threshold of this material. (See Figure 40.). The humidity-saturated specimens exhibited more severe delaminations but less charring than the dry specimens. (See Figure 41.) Figure 42 shows the progressive degradation of system 14 with each successive flash. The right laminate shows blistering and delamination after three flashes, while the left laminate shows charring and delamination after five flashes at 20 cal/cm².

25 CAL/CM²

All system 14 composites, both dry and humidity-saturated, suffered catastrophic delamination at the 25 cal/cm² energy level. Some of these delaminations were so severe that the face sheet was blown into the quartz lamp bank with such force that it caused several thousand dollars in damage.

30 CAL/CM²

No system 14 composite testing was performed at the 30 cal/cm² energy level due to the severe failure mechanisms which occurred at the 25 cal/cm² energy level.

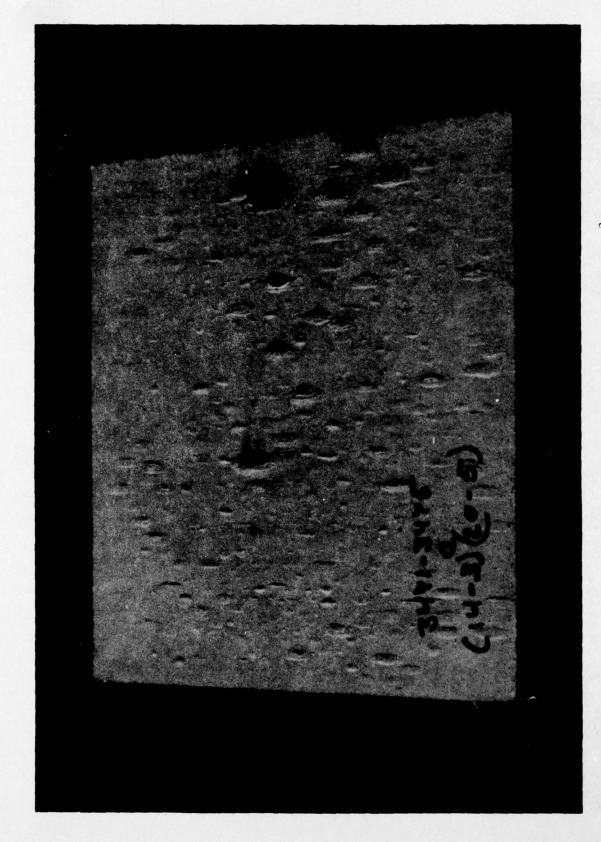


Figure 40. System 14 - threshold damage at 20 cal/cm².

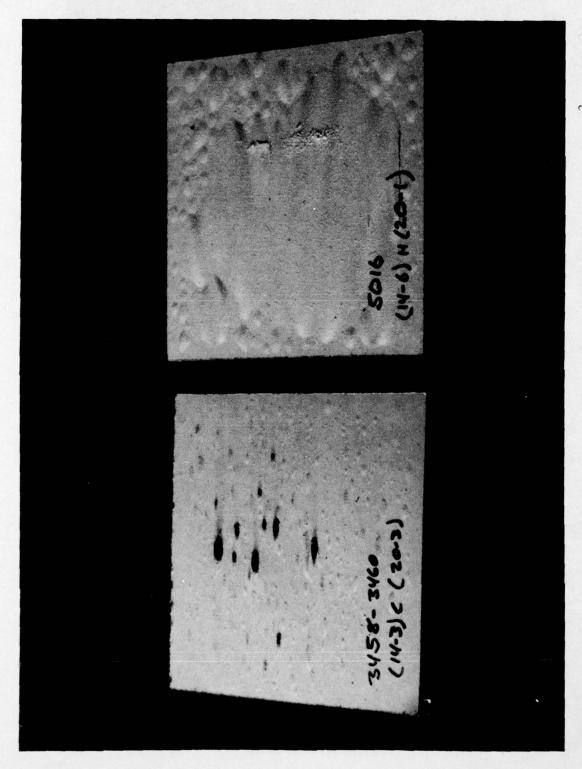


Figure 41. System 14 - humidity saturated vs dry specimen at 20 cal/cm².

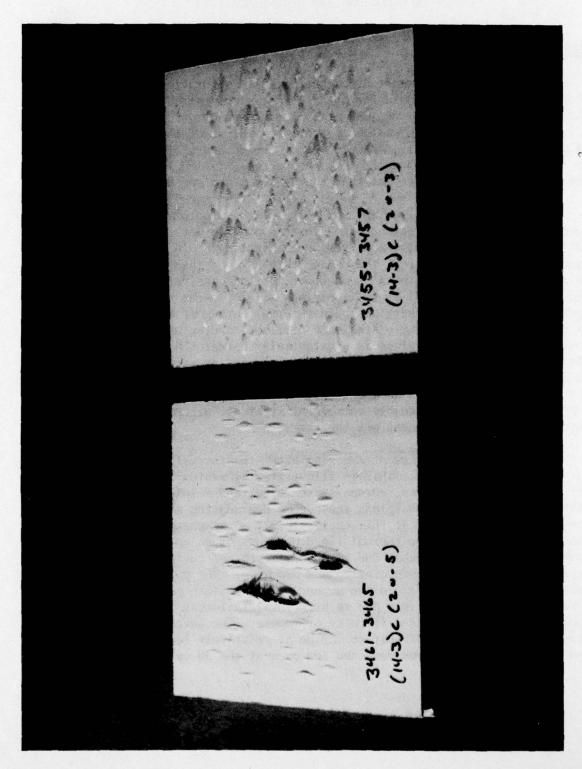


Figure 42. System 14 - three vs five flashes at 20 cal/cm².

Section IV

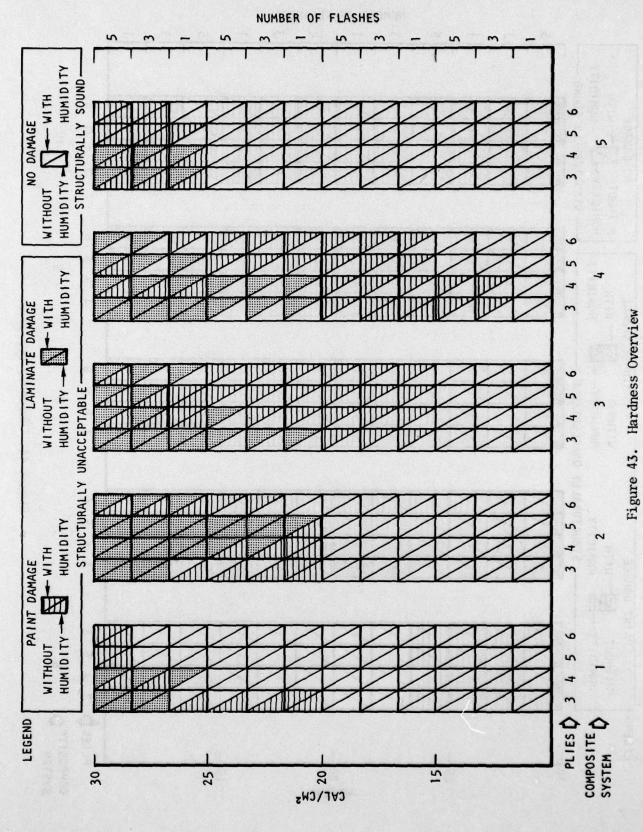
CONCLUSIONS AND RECOMMENDATIONS

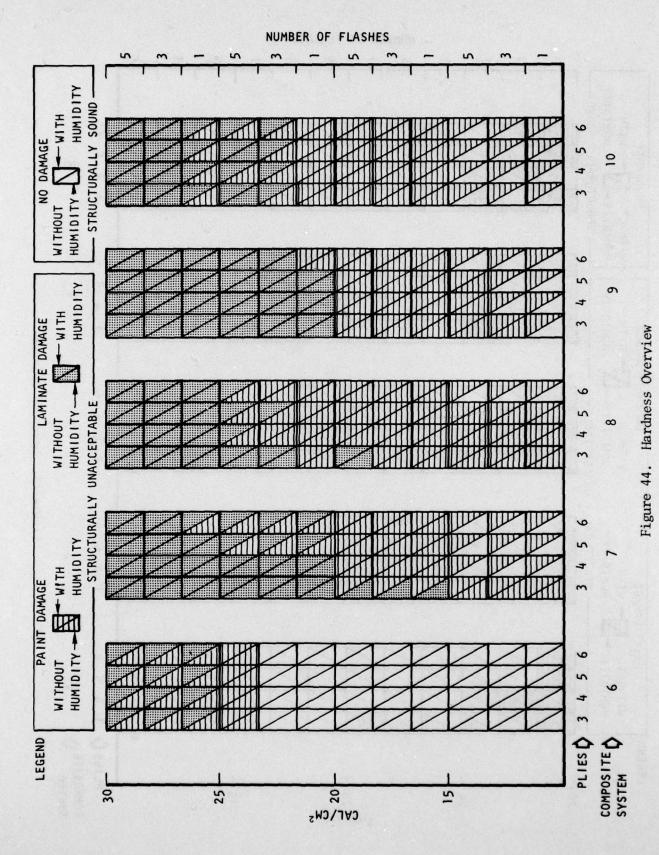
CONCLUSIONS

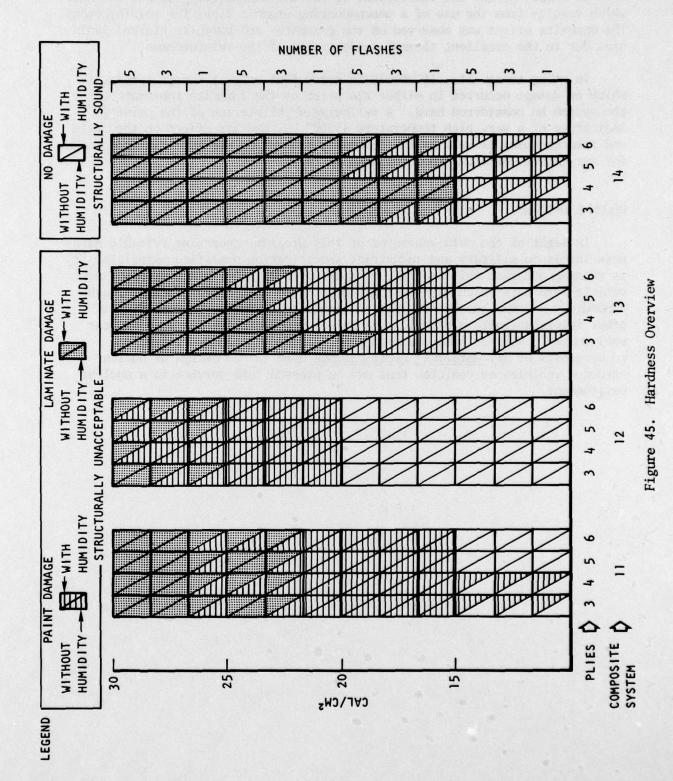
This program has demonstrated that humidity saturation substantially reduces the damage threshold for organic matrix composites exposed to the thermal pulse produced by the endoatmospheric detonation of a nuclear weapon. This hardness degradation was consistent, regardless of the initial hardness of the composite material. An overview of the hardness of these 14 materials is shown in Figures 43, 44, and 45. An open or unfilled triangle indicates the specimen was unaffected at that energy level and can be considered hard.

This test program illuminated a very serious problem with organic matrix composites. All epoxy resins tested in this program met the requirements of MIL-R-9300B, "Resin, Epoxy, Low-Pressure Laminating." In spite of military specification qualification, the damage threshold of these resin-matrix materials, when reinforced with the same fiber, varied considerably. For example, the F-161 epoxy with 7781 style fiberglass reinforcement and the CE-9000 epoxy with 7781 style fiberglass reinforcement both meet MIL-R-9300B, as well as being qualified to many of the same airframe manufacturer's specification; however, the F-161 fiberglass is substantially harder than the CE-9000 fiberglass. The four-, five-, and six-ply F-161 laminates were unaffected at 25 cal/cm², while the CE-9000 laminates suffered catastrophic resin degradation and laminate delamination at the 25 cal/cm² energy level. These two systems have been used interchangeably on several strategic aircraft based on the assumption that the hardness was the same.

A change of reinforcement can also alter the thermal hardness threshold for a given resin matrix. This is illustrated by system 1, an F-161 epoxy reinforced with 7781 glass, versus system five, and F-161 epoxy reinforced with 181 quartz. The fiberglass shows some degradation at 25 cal/cm², while the quartz is unaffected at this energy level. A graphite reinforcement substantially reduces the thermal flash hardness of an epoxy or polyimide composite. This is probably due to the differential coefficients of thermal expansion which cause resin crazing and cracking. The epoxy/boron composites tested all failed in an explosive manner. This failure mechanism could cause severe problems in the field due to structural failures, increased drag in secondary structures, and difficulty of repair. The Kevlar-reinforced laminates suffered substantial surface damage at relatively low energy levels but the back face temperatures remained low even at the 30 cal/cm² energy level.







This is caused by the low coefficient of thermal conductivity of the laminate, which results from the use of a nonconducting organic fiber for reinforcement. The opposite effect was observed on the graphite- and boron-reinforced laminates due to the excellent thermal conductivity of the reinforcement.

In using these data, it should be noted that only at energy levels at which no damage occurred in either the paint or the laminate substrate can the system be considered hard. A yellowing or blistering of the paint is indicative of a very high temperature (>500° F), and its effect on the resin and reinforcement as well as the composite properties are essentially unknown for very-short-duration exposures such as these.

RECOMMENDATIONS

In light of the data generated on this program concerning variable hardness levels on military and industrial specification qualified materials, it is recommended that this program be expanded to consider other exterior air vehicle materials which may react differently to high-energy thermal flash exposure. A comprehensive evaluation should be conducted to determine if other interchangeable air vehicle materials react differently in a nuclear environment. Nuclear hardness should be included in the qualification requirements of <u>all material specifications</u> used in the design of nuclear-hardened vehicles or vehicles that may be pressed into service in a nuclear environment.

All testing was performed at the DNA-Service Thermal Radiation Test facility, Wright-Patterson Air Force Base, Dayton, Ohio. The facility is operated by the University of Dayton Research Institute under the direction of the Defense Nuclear Agency Headquarters, Washington, D.C. The facility and its operational capabilities are described in the Test Procedures Handbook UDRI-TR-77-28. The approximate pulse shape used during this program is shown in Figure A-1. The following is a brief description of the facility.

QUARTZ LAMP BANKS

The intense radiation needed to simulate a nuclear flash can be produced by a series or bank of tungsten filament, quartz lamps. Two banks are available in the facility; they are designated the Stationary Quartz Lamp Bank (SQLB) and the Mobile Quartz Lamp Bank (MQLB). (See Figure A-2.) The SQLB is primarily used for instrumentation check-out and radiation-only exposure tests. The MQLB is used on conjunction with the simulation of aerodynamic or mechanical loads.

The large bank area produces a one-dimensional radiation source, approximately 6 by 5 inches. The incident radiation on a test specimen is a function of the distance from the bank source. Certain tests require protecting the lamps; this is normally accomplished by inserting a quartz window between the lamps and the exposed specimen. The quartz window absorbs a portion of the radiant energy, thereby slightly reducing the incident radiation.

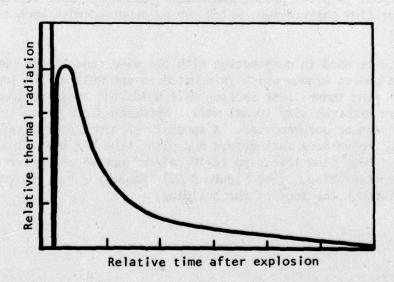


Figure A-1. Pulse shape.

One each thermal temperature closed-loop controller and data-trak controller are available for making minor adjustments to the radiation history.

During testing, lamp voltage and current are normally monitored, allowing a confirmation of the radiation produced if the actual radiation flux cannot be monitored during the test. The heat flux for a specified condition is repeatable to within ± 1 percent.

Several radiometers are available for determining incident radiation flux as a function of time. Response times are on the order of 100 milliseconds for the radiometers. Radiometer output is typically recorded on x-y plotters.

Although the one-dimensional heating area is restricted to approximately 6 by 5 inches, no physical constraints are placed on the maximum test specimen size. However, care should be taken to minimize edge heat losses on larger specimens.

AERODYNAMIC LOAD SIMULATION

An open-circuit pull-down wind tunnel is available to simulate aero-dynamic flow over specimens exposed to high-intensity radiation. The wind tunnel is shown in Figure A-3, and the test section in Figure A-4. The 12-inch-long test section has a 15/16 by 4-1/2 inch cross-sectional area. The constant free-stream velocity is nominally 790 feet per second, and the nominal mach number is 0.7. The Reynolds number based on the inlet wall le length can be varied by 2 by 10^6 to 18 by 10^6 , depending upon which inlet section is used. A pitot probe, manometers, and a pressure transducer are available for flow calibration, which can be supplied with each test program, as required.

The MQLB is used in conjunction with the wind tunnel; the beam is brought in through a quartz window which is mounted in one wall of the test section. The opposite wind tunnel test section wall holds the test specimen, which is mounted flush with the wind tunnel wall. Specimen sizes up to 4.500 by 3.970 inches can be accommodated. A special "specimen plate" is available for mounting the various radiometers and pitot tube for heat flux and flow calibration. Heat flux levels up to 40 cal/cm² per second are readily achieved with this configuration. (See Figure A-5.) Exhaust gases are vented to the atmosphere through the roof of the building.

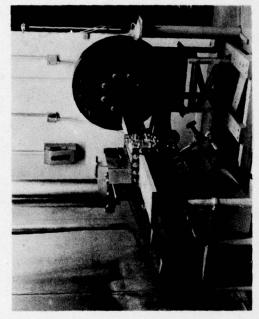


Figure A-3. Wind tunnel.

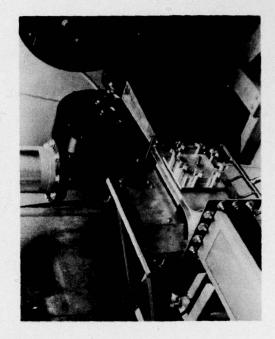


Figure A-5. Specimen plate.

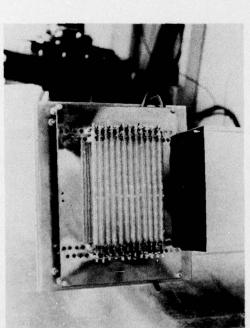


Figure A-2. Quartz lamp bank.

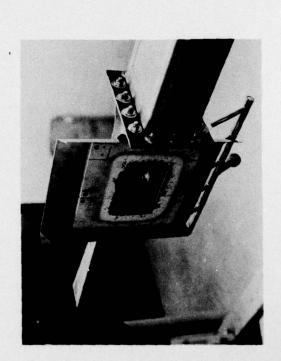


Figure A-4. Test section.

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